CRITICAL LITERATURE SURVEY OF MISSILE BLAST RESISTANT MATERIALS

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DEPARTMENT OF CERAMIC ENGINEERING
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U. S. ARMY ENGINEER DIVISION, OHIO RIVER
CORPS OF ENGINEERS
OHIO RIVER DIVISION LABORATORIES
CINCINNATI, OHIO 45227

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FORWARD

This survey reported here was conducted in the Department of Ceramic Engineering, University of Illinois, Urbana, Illinois, under a cooperative agreement with Ohio River Division Laboratories, U. S. Army Engineer Division, Ohio River, Cincinnati, Ohio as a part of an overall program relating to launch facilities operated by John F. Kennedy Space Center, National Aeronautics and Space Administration, Advanced Studies Office, Facilities Engineering and Construction Division. Work was performed during the period June 1, 1964, to August 1, 1965.

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Appreciation is also expressed to the various companies, government agencies and laboratories who so kindly supplied background material for the survey.

At the time of the preparation of this report W. L. Augur and F. E. Richter were Research Assistants (half time) in the Department of Ceramic Engineering, University of Illinois. A. W. Allen is Professor, Ceramic Engineering at the same institution.

ABSTRACT

A literature survey was conducted to study the criterion for the utilization of refractory materials for thermal protection of launch facilities. Sources were surveyed from the viewpoint of the refractories engineer including: (1) published literature relating to refractory materials in general; (2) literature relating to specific applications under simulated or actual launch conditions; (3) preliminary contacts with refractories manufacturers and engineering departments; (4) preliminary contacts with government agencies and facilities working in this or related problem areas. A historical review traced the development of the subject from the early use on aircraft runway pavement to present missile sites. Kennedy Space Center inspections and Ohio River Division Laboratory Programs were used as examples to characterize the problems involved.

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I. INTRODUCTION

As launch vehicles for the space program have increased in size and thrust, problems of maintaining the launch pad structure and protecting the surrounding facilities structures from thermal radiation have become major considerations. The thrust to weight ratios characteristic of contemporary launch vehicles have imposed severe heat loadings in combination with vibration, flame impingement, and particle impact. The application of refractory materials has progressed in scale beyond a simple system composed of a single refractory concrete pad surrounding a steel deflector placed in the primary impingement zone. In fact unprotected metallic deflector plates of any configuration are not usually adequate.

The utilization of refractory materials in this type of environment becomes one of developing a multiple materials system for the special conditions imposed. This design function must be supported by an understanding of the response of the materials to the expected environment. This is gained from (1) simulative tests and (2) evaluation of materials after a service tenure. Hopefully, the combination of these will produce detailed requirements in terms of realistic materials specifications and improved design criteria.

It is also true that the extent to which refractory materials are used in a launch facility now makes their placement a matter for careful engineering planning from design to installation. This is a situation quite similar to the application of refractories in the process industries where close cooperation

between producer and consumer makes the venture mutually profitable. The explosively changing pattern of utilization of refractories in future facilities requires an even more careful cooperation at all levels of planning whether it be a launch facility or a facility for one of the process industries.

It is the purpose of this survey to review these problems from the viewpoint of the refractories technologist using: (1) published literature relating to refractory materials in general; (2) literature relating to the specific application; (3) preliminary contacts with refractories manufacturers and engineering departments; (4) preliminary contacts with government agencies and facilities working in this or related problem areas.

Information collected has been referenced in the Appendix of the report under section C listed alphabetically by author and subdivided into appropriate sections. This same bibliography has been prepared in the form of a card set adapted from the ASM-SLA Metallurgical Literature Classification. A more sophisticated system was not considered feasible because of budgetary limitations. In fact the entire retrieval subject is still in such a state of change that it was not considered expedient to dwell on adapting the information herein to an existing system. The card system employed does allow for periodic updating.

II. HISTORICAL BACKGROUND

With the advent of jet assisted takeoff and jet propelled aircraft in the 1940-1950 decade, runway and maintenance area runup pavement deterioration became a problem due to the impingement of

exhaust gases. Surface impingement and heat flux patterns in paving media resulted from the inclined attitude of the engine. Early investigations were concerned with defining the temperature distribution in conventional paving media, namely bituminous pavements and portland cement concrete pavements. The local distress in the paving was shown to be dependant on the temperature and exhaust velocity of the gases and the duration of exposure but was particularly sensitive to the attitude of the engine relative to the paving surface. Bituminous pavements were generally satisfactory only when the surface temperature was less than 300°F and the blast impingement angle did not exceed 6 degrees to the horizontal. Softening point (ASTM) of paving grade bitumens also allowed damage to pavements due to vehicular traffic over surfaces whose temperature had exceeded 300°F.

Rigid pavements were generally not damaged by jet operations when the surface temperature was below 1000°F. This temperature limitation was to a degree dependant on the type of aggregate. By avoiding the use of aggregates in which the quartz was the primary phase and employing basic igneous rock, non-glassy blast furnace slag or clay-based manufactured aggregate the temperature could be extended to the 1500°F range.

Investigators realized, however, that if the entire body of pavement reached this temperature, the temperature stability (dehydration) of the portland cement bond and perhaps even reactions between the cement and the aggregate would be the limiting factors. A library study on the effects of high temperature on concrete by the Ohio River Division Laboratories of the Corps of

Engineers had defined these limitations as early as January, 1947. In the range of 1600°F the cement bond deterioration would completely destroy the concrete while even at 1100°F serious reduction in strength would occur. This meant that a solution to the problem could no longer be approached by modifying the aggregate or adjusting the other parameters of conrete mix design such as cement-aggregate ratio, aggregate size distribution, and water content.

Consequently a program to study the properties of refractory brick and the product line of calcium aluminate bonded refractory concretes referred to in the trade as "castables" was started. A second library study in April of 1947 recommended that materials such as zirconia, magnesia, silica, mullite and forsterite be adequately tested. Unfortunately this survey placed entirely too much emphasis on melting point because that seemed to be the principal basis on which refractory classes were selected. Further testing of refractory concrete under the guidance of manufacturer's recommendations was also proposed.

At the end of this decade (1940-1950) and in the beginning of the next (circa 1951-54), the problem of jet engine exhaust impingement became less important because the attitude of most jet aircraft being designed became such that the engine was moved to a location considerably higher above the pavement and/or the exhaust was essentially parallel to the pavement. In the interim attention was switched to a detailed time-movement study of operational aircraft including starting, taxiing, preflight, takeoff, landing and maintenance runs. Attention thus was devoted to long

time duration performance of pavements under less severe impingement conditions. Jet engines with sustained afterburner operation during stationary (maintenance runup) operation produced paving surface temperatures in excess of 2000°F. Refractory concrete was definitely indicated for selected areas.

The engine attitude change has now come full circle because the development of vertical takeoff and landing (VTOL) vehicles with jet engine power plants will produce an impingement at essentially 90° (excluding helicopter vehicles, of course).

The beginning of the 1950-60 decade saw the appearance of work relating to rocket exhausts. The impingement problem was again paramount since all launchings were necessarily made at high impingement angles with some at close to 90°. The combination of sonic or supersonic exhaust velocities with very high flame temperatures produced stringent requirements on materials comprising launch facilities.

Early in the missile program it was realized that exhaust impingement on flat surfaces, whether ceramic or metallic would result in extreme cavitation. The general problem then would have to be approached by a combination of studies of blast deflection configuration and resistance of materials.

By the middle of the second decade (1956) work had lead to the following results:

(1) portland cement concrete was inadequate for critical impingement areas subjected to temperatures above 1500°F;

(2) evaluation of aggregates to be used with calcium aluminate cement in which thermal shock was the basic criterion for selection;

(3) construction of a minature rocket assembly near ORDL to simulate jet and rocket blasts;

(4) tests to determine heat and blast resistance of selected formed refractories;

(5) evaluation of commercially available castable refractories placed by pneumatic gunning or normal concreting methods;

(6) preparation of a specification to provide a refractory material resistant to heat and blast on airfield pavements and rocket launching facilities;

(7) field test installations (Patrick and Edwards AFB) to correlate laboratory data with service conditions as well as defining placement problems;

(8) realization that the configuration of metallic deflectors in the direct impingement zones was a factor to be studied.

A deflector based on a curved plate was developed for use in conjunction with the early Redstone-Jupiter missiles. This deflector, called the "Mexican Hat," was pyramidal in shape, consisting of 1/4 inch cold rolled steel with a replaceable solid tip. It was placed directly under the missile and deflected the exhaust horizontally along a castable refractory pad. Regular concrete surrounded the castable section and was also used for the base of the pad. Launching from this type of arrangement proved acceptable even though some erosion of the castable refractory around the metal reflector was noted. (Figure 1)

Table I shows the general historical development of the participation of ORDL during the 1950-1960 decade with reference to specific missile programs.

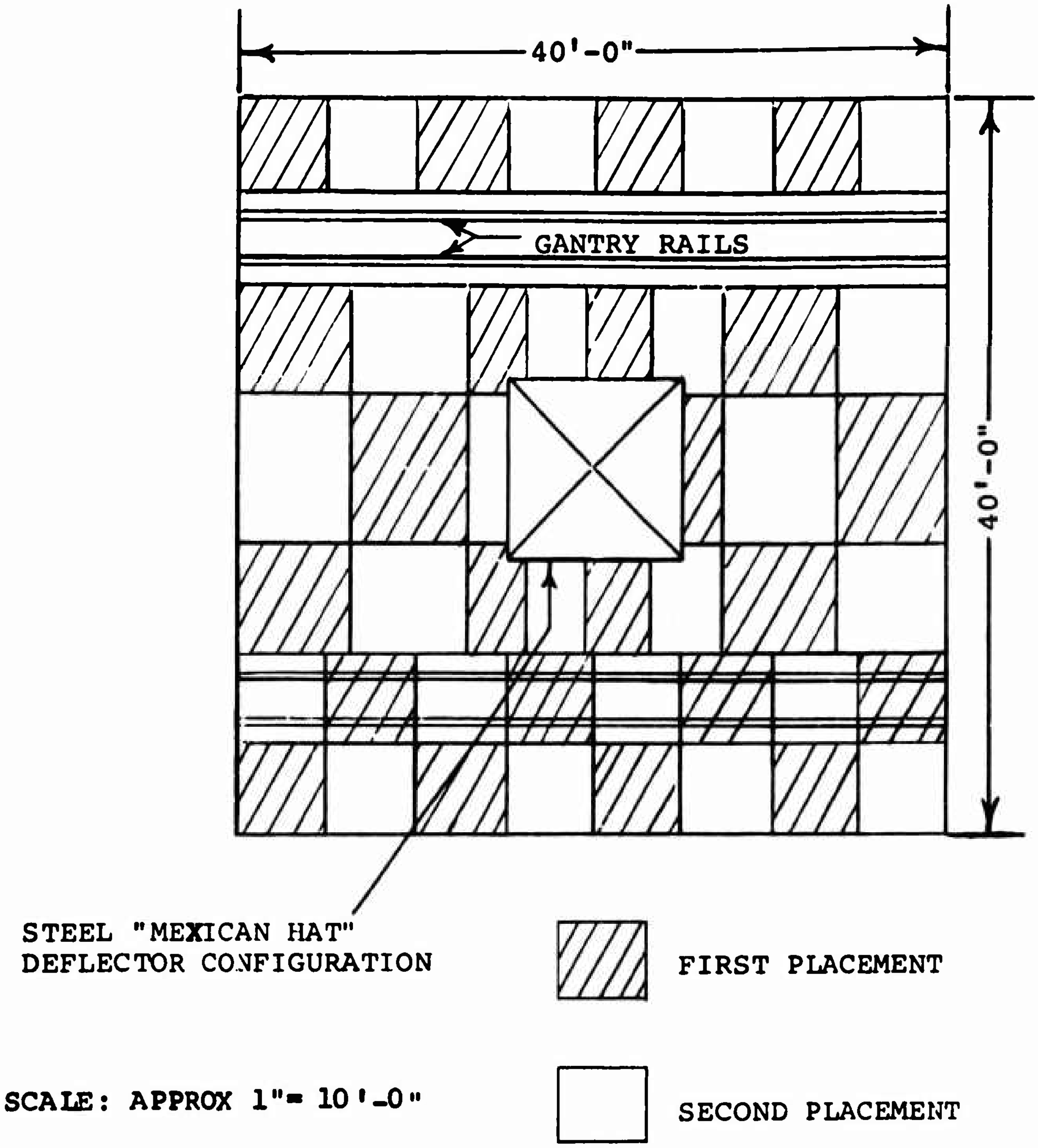

FIGURE 1. Early Configuration of Launch Pads for Redstone-Jupiter Missile (1956 Courtesy ORDL)

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TABLE I

Year	Projects
1951	Feasibility Study, General.
1952	Feasibility Study for Honest John and Terrier. Initial concept of deflection.
1953	Small-scale tests for Honest John and Corporal tactical deflectors. Deflector materials tests, small scale.
1954	First full-scale static deflector tests (Honest John). Design of full-scale deflector for Corporal.
1955	Corporal full-scale deflector tests. Full-scale materials tests. Design and full-scale test of stabilizing deflector for Nike-Ajax. Redstone and Nike B small-scale deflector tests. Correlation with Navaho deflector tests.
1956	Utilization of blast forces to stabilize Honest John and Littlejohn lightweight launchers. Model tests for Redstone and Jupiter tactical deflectors. Camouflage tests for Nike-Ajax. Model tests for aircraft blast fences
1957	Model tests and first full-scale tests for aircraft blast fences. Model tests for Redstone and Jupiter tactical deflectors for lightweight launchers. Model tests for Nike Hercules, Nike Zeus and Hawk.
1958	Model tests for Redstone and Jupiter unprepared site launchings. Model tests for Nike Hercules and Nike Zeus cellular concepts. Model tests for Hawk Offshore System, Full-scale aircraft blast fence tests. Full-scale Hawk tests. Full-scale Atlas deflector tests.
1959	Jupiter full-scale tests on unprepared sites. Model tests for Nike-Zeus cellular structures. Model and full-scale tests for Hawk. Full-scale aircraft blast fence tests. Data exchange with Air Force, Navy and NASA.
1960 July-August	Mace model tests and theoretical correlation.

Per annum funding for this program started at about $1000 and increased to the $60,000 range as the number and engineering sophistication of rockets increased.

As early as 1954, tentative specifications for "refractory castable mix" were written. These were based on a combination of: (1) American Society for Testing Materials Committee C-8 on Refractories test procedures; (2) Corps of Engineers procedures; (3) special high temperature thermal shock and spalling tests utilizing a minature rocket developed at ORDL; and (4) sampling, curing and placement requirements resulting from field trials and previous experience with paving concretes. The significant high temperature requirements included permanent volumetric change limitations (not more than minus 1.0 percent after 5 hours at 2000°F.); pyrometric cone equivalent (not less than cone 15); room temperature transverse strength (800 p.s.i. after 5 hours at 2000°F.) room temperature crushing strength (2200 p.s.i. after heating to 2000°F.); and a thermal shock and spalling limitation ("minor surface melting without spalling") resulting from the minature rocket test where the angle of flame impingement, instantaneous surface temperature and the duration of exposure were varied to relate to correlative conditions expected in service (Figure 2). It is interesting that a freezing and thawing requirement was also included.

The specification was modified a number of times as a result of further field experience and cooperation with refractory suppliers. Its development included more realistic strength requirements in that crushing strength was eliminated and modulus of rupture minima were based on the temperature to which the cured

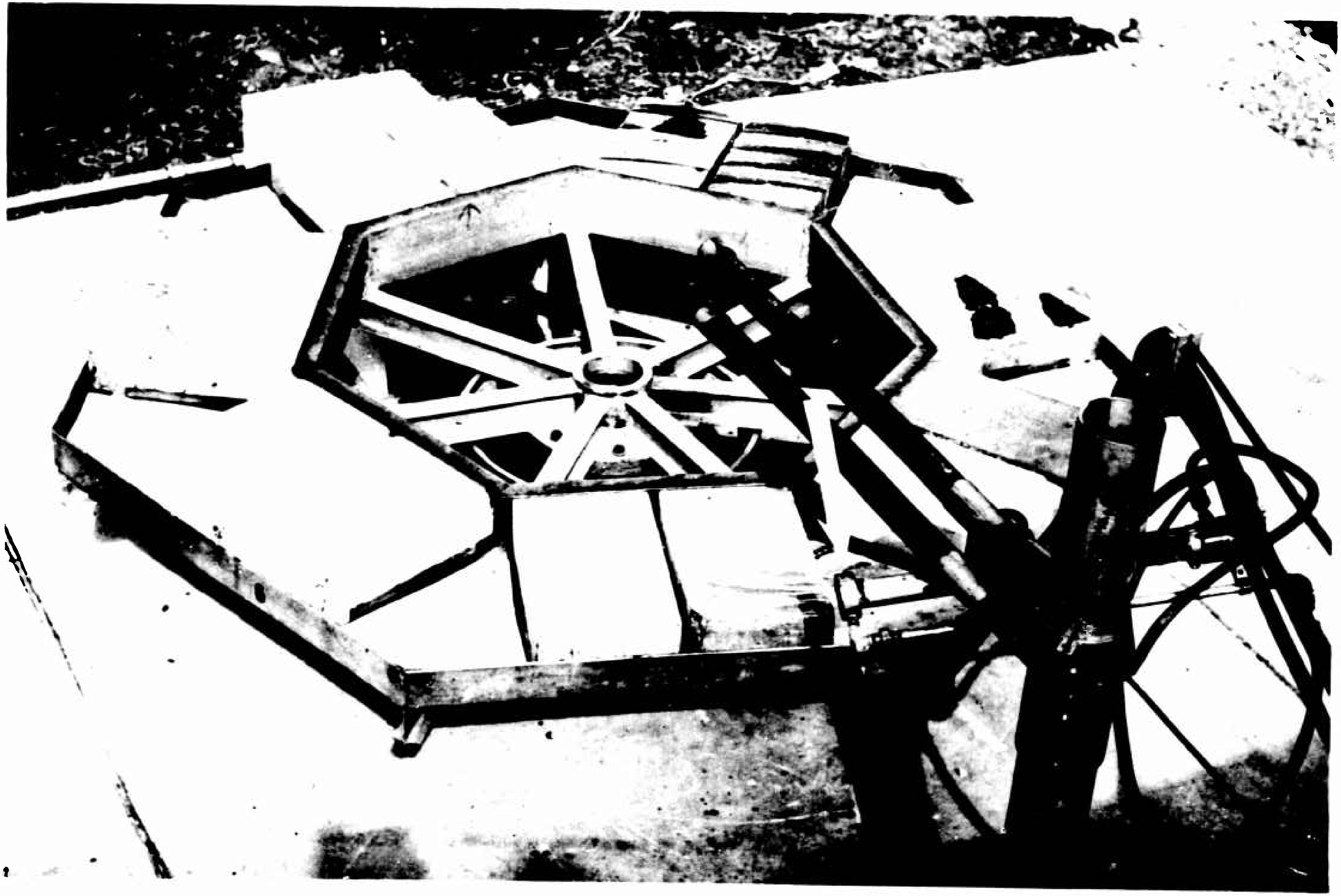

FIGURE 2. Minature Rocket Test Facility Showing Rotating Material Test Stand and Rocket Positioning Assembly. (Courtesy ORDL)

sample had been heated (e.g. 500 p.s.i. after heating to 2000°F. and cooled to room temperature). Pyrometric cone equivalent was excluded as not being representative of the bulk properties of the material. The material class for which this specification was developed had a specific definition of "castable" in terms of a refractory aggregate and a "hydraulic bonding medium." Calcium aluminate cement bonded aggregate was obviously the class of material intended. It recognized also materials applied by gunning. By 1958 the use of this type of material had been practiced in the field to the extent that placement became a prominent part of specifications under "Notes to the Contracting Officer." Placement carried definite requirements with respect to: (1) anchoring the castable surface to the portland cement concrete base with welded wire fabric and 1/2" x 8" anchor bars; (2) preparation of the underlying interface with an epoxy resin adhesive to bond the castable to its base; (3) a placement sequence in alternating 4.0 x 4.0 foot squares (usually 3" thick) to allow for the characteristically high heat of hydration of calcium-aluminate cements during curing; (4) curing procedures consistent with this type of cement and (5) finishing procedures adapted to the refractory concrete. (Figure 3)

Overlapping the time of this development at the close of the 1950-1960 decade the environmental conditions imposed by rockets of lower thrust to weight ratio indicated that the surface temperature and severity of impingement would require a refractory material not limited by the thermal degradation of the calcium aluminate cement. This could only mean refractory brick paving trials. As rocket launch systems, therefore, began to improve in efficiency from a thrust-to-weight ratio of about 4.50 to 1.30 or less, the

FIGURE 3. Placement of Refractory Concrete Pavement at Early Redstone-Jupiter Site. (Note Squares for Alternating Placement of Gunned Material; Courtesy ORDL)

thermal containment problem was exaggerated. The somewhat longer "period of dwell" prior to liftoff also began to impose consideration of the thermal protection of facilities adjacent to the pad structure.

The use of water as a cooling medium became part of the operation to expedite the testing of rocket engines both in static test sites and launch areas. The ORDL specification for refractory concrete recognized usage limitations by specifically stating that the material was intended for dry operation where no water would be encountered except that due to normal rains. It was recognized that calcium aluminate cement bonded materials required the hydrated bond to remain structurally integrated. Facilities located in semi-arid or arid regions might therefore introduce low humidity climatic conditions which in combination with high temperature could cause the concrete to loose strength during aging. This indicated again the need to study a broader class of refractory materials systems.

This was all dramatically illustrated by a static test facility at Edwards Air Force base. A deflector was formed by excavating an igneous rock zone below the rocket exhaust and covered with a gunned refractory concrete. The rocket engine was positioned above the deflector on the water cooled holding pad. Initial firings of the rocket destroyed the deflector in the immediate area of exhaust impingement which was 183 feet from the rocket nozzle. The test section destroyed was replaced by a test panel of refractory brick bonded to the surface with an epoxy resin, coated with calcium aluminate, and the surface ground to specifications.

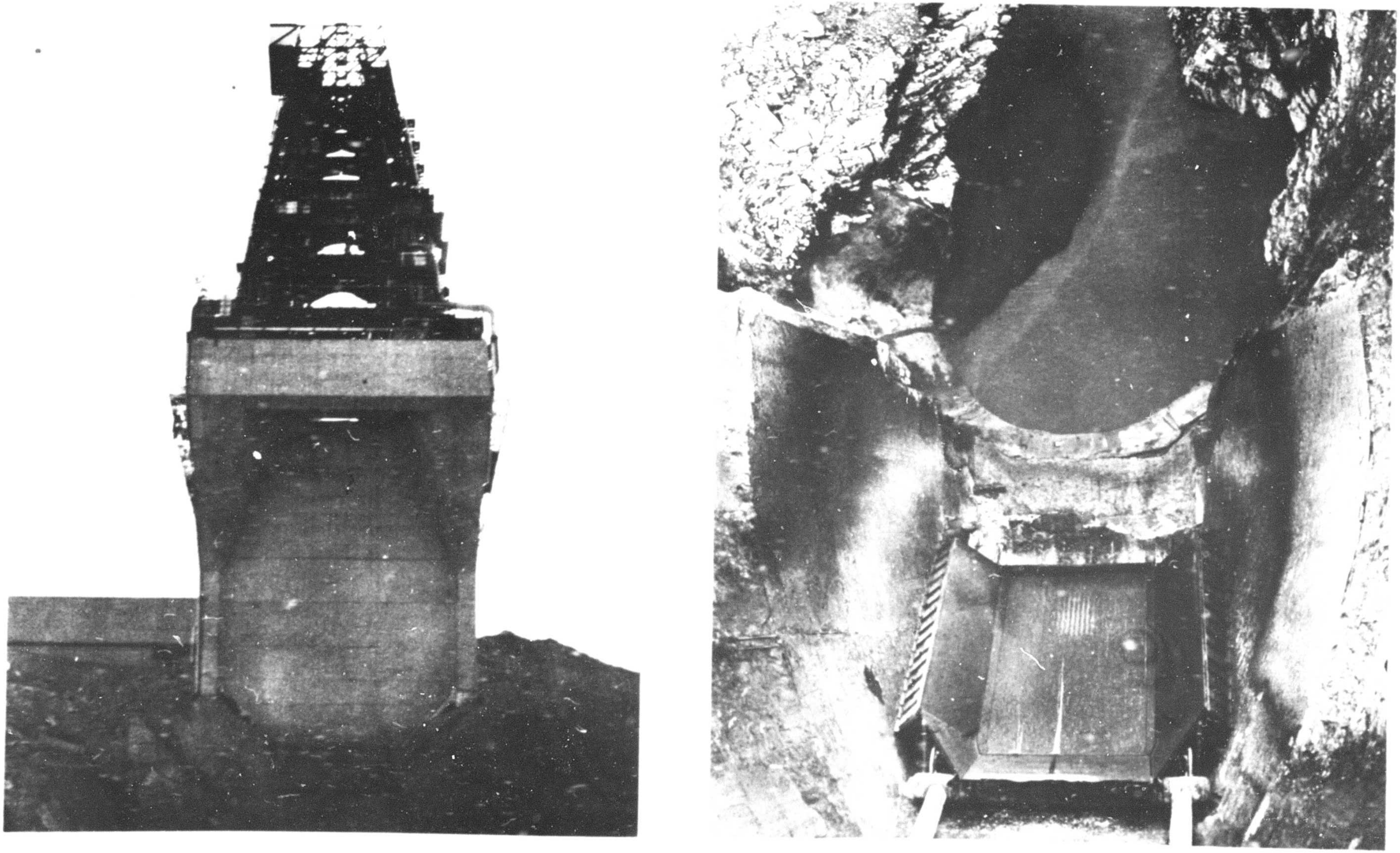

FIGURE 4. Static Rocket Test Site, Edwards Air Force Base, Showing Extreme Cavition in Igneous Rock Base Due to Exhaust Impingement. Water Cooled Steel Deflector Plates Shown in Right Photo. (Courtesy A. P. Green Fire Brick Co. and Rocketdyne Division, North American Aviation, Inc.)

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Surface grinding was necessary because a slight ridge in the network would cause melting and brick spalling from the increased exhaust velocity over the ridge. This experience reiterated that the following major problems would always have to be considered: (1) water seepage from cooling the holding pad; (2) impingement angle of the exhaust; (3) economics and time involved in installation and maintenance of the refractory deflector portion of the facility. (Figure 4) It was not considered feasible to shut down the test facility because the vital ICBM program would be delayed. ORDL conducted a crash program for testing refractory brick in which ten 4 1/2" x 2 1/2" x 9" refractory brick from each participating producer were laid in regular bond course and subjected to a 300 second exposure of the miniature rocket. Blast cycles developing surface temperatures of 3000°F at a 45° impingement angle were imposed on each panel in the as-received condition and after immersion in water. These brick panels were laid over a base pavement block with thermocouples at the interface to establish base pavement temperature rise. Unit weight, percent water absorption and saturation coefficients were determined for each type of refractory. The effectiveness of an epoxy resin was also evaluated by determining the shear stress applied to a prepared joint between the test refractory and a portland cement concrete prism bonded to the 2 1/2" x 9" face of the refractory. Several castables were also evaluated.

It is interesting to note that one of the limitations on refractories for this test program was availability. Actually only six manufacturers responded in the time available (approximately

a two week period).

These results led to a recommendation for a test panel consisting of two types of super duty fireclay brick, a mullite refractory and a silicate bonded silicon carbide refractory. From the cost analysis of this panel it is worthy to note: (1) the super duty fireclay refractory was roughly one tenth the cost of silicon carbide; (2) the mullite refractory represented a premium in cost of about five times that of the fireclay; (3) the epoxy adhesive was almost twice the cost of the fireclay refractory and (4) installation of refractories and preparation of the damaged area represented about half the total costs. These facts are mentioned here to point out the importance of treating the refractory protection system as an engineering problem as well as a materials selection decision.

At Cape Kennedy Complex 36A used for launching the Atlas-Centaur missile, a refractory concrete flame bucket deflector and a refractory concrete trough to carry away the cooling water was damaged extensively from flame impingement and the spalling of wet concrete subjected to high temperature. This is discussed in more detail later in the report.

By 1958 a tentative specification had been developed which recognized super duty fireclay brick (ASTM C-27) as an operational refractory. It was a combination of Federal Specifications, American Society for Testing and Materials Committee C-8 classifications and test procedures and Corps of Engineers test methods for concretes. It recognized an adhesive ("organic mortar") of the epoxy resin-polysulfide polymer type with a suitable curing agent and inert mineral filler (silica or mica). To the ASTM

criteria for classifying super duty spall resistant fireclay brick were added dimensional tolerances, modified reheat requirements, average coefficient of thermal expansion limit, freezing and thawing resistance and high order thermal shock resistance as determined in the ORDL rocket test. Details of preparation of the epoxy resin were carefully described.

Placement was emphasized as to: (1) smoothness of base pavement; (2) placement of epoxy mortar; (3) laying brick; (4) sealing joints; (5) grouting with calcium aluminate to fill joints and smooth the surface to produce a variation not exceeding 1/16". Because of the possible toxicity of resins and curing agents, safety precautions during handling were reiterated.

The specification also distinguished between paving for jet aircraft and paving for rocket motor operation based on surface temperature, impingement angle, duration and number of cycles in the rocket test.

In the present 1960-1970 decade this specification was developed for silo linings for critical areas of missile facilities (1960) and finally in the present form as a Guide Specification for Military Construction Pavement, Refractory Brick for Critical Areas of Aircraft and Missile Facilities, (ORDL January 1965). In these later specifications the details of all aspects of defining, classifying, testing, placing, and evaluating refractory brick in a refractory system designed for the launching pad in an operation where large quantities of cooling water are employed have been further refined.

In the present system for the Saturn launch site, the small "Mexican hat" deflector has now been replaced by a massive

knife-edge deflector made of cold rolled steel and protected by a thick coating of special refractory castable. Super duty fireclay brick satisfying the 1965 specifications form the pad. They are placed with the epoxy adhesive and smoothed with neat calcium aluminate cement to form a monolithic surface. The environment includes high volume flow of cooling water.

Thus during the current decade (1960-1960) blast protection based on a knife edge deflector with castable protective surface and a super duty fireclay brick water-cooled pad is an operational system for liquid fueled rockets.

In the middle of this decade (1963-1966) an entirely new set of environmental conditions relating to the use of solid fueled rockets is being encountered. Application of refractory protective systems is thus starting a new round of materials testing. Solid propellant rocket motors in a configuration designed to simulate launch conditions on a subscale basis is the current approach. (Figure 5) The complexity of these simulative systems introduces much needed work relating to the operation of the facility in addition to materials evaluation. Thus theoretical studies to define engine characteristics, exhaust jet properties, temperature and pressure distribution, and flame radiation characterization typifies these programs.

Because solid particles of alumina are present in the exhaust gases, the characterization of the particle as to size, distribution, and state of solidity become important. The presence of the hard particle also introduces the study of multiparticle impact on hot protective materials, the particles often moving at supersonic velocities.

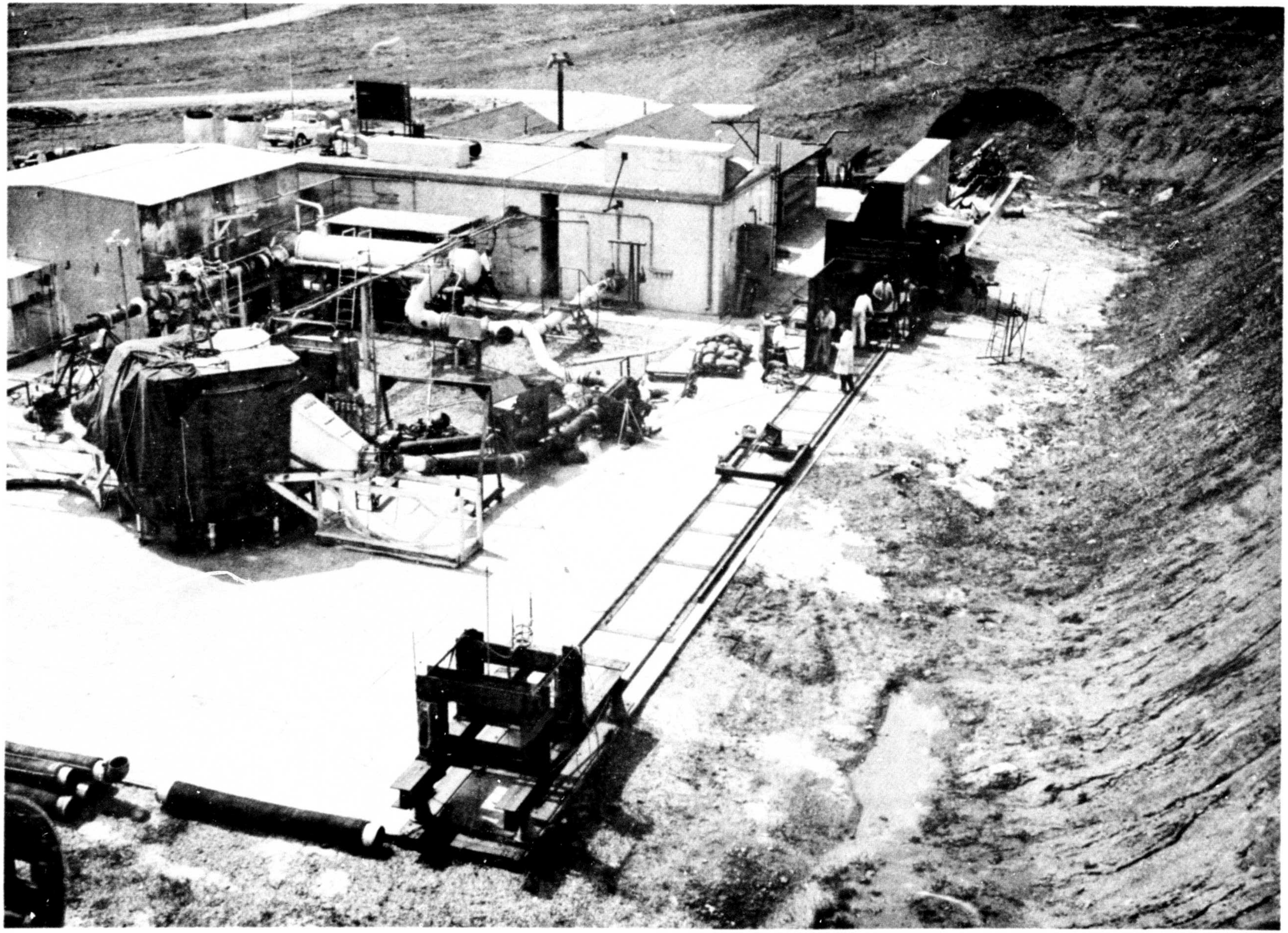

FIGURE 5. SPREE Simulated Launch Test Site and Associated Facilities. (See Section V for Details. Courtesy Aerospace Division of Martin-Marietta Corp., Denver, Colorado)

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The "new environment" is expected to include: (1) stagnation temperatures of 5000°F; (2) stagnation pressures up to 750 p.s.i.; (3) erosive particle streams traveling at supersonic velocities; (4) combustion products involving highly reducing gases (H_2, CO, H_2S, nitrogen oxides); (5) combustion products producing corrosion (HCl, H_2S, nitrogen oxides); (6) high order thermal shock; and (7) mechanical vibration and shock. The protection system actually takes on many of the aspects of materials development for space vehicle thermal shields and nose cones. Refractory technologists are therefore looking at materials for protection which imply a portion of the system being sacrificial with emphasis on periodic maintenance and economical repair.

Blast control structures will begin to assume a complex configuration beyond that of the pad concept, particularly if hardened sites are considered. Deflectors, exhaust tunnels, blast diffusers and heat radiation shields may all become a part of a future refractory materials system for blast control. The economical placement of materials in this system may require all types of refractories from the ablative, low density, fiber-resin composite to dense, hard, high melting point, low expansion oxide refractories. Materials applied by bulk placement (castables and gunned materials) as well as refractory brick structures will all certainly have their places. This is evidenced by the wide variety of ablative plastics to very hard materials in the carbide family being studied. Refractory castables of the calcium aluminate base variety, phosphate bonded oxides, refractory metals, low expansion ceramics and a variety of specially prepared composites complete

the spectrum. Although some of this work might be considered repetitive, too exotic and even naive to a refractories engineer, it illustrates the wide range of approaches needed to solve the problems where the orders of magnitude of thermal-mechanical requirements are so far beyond any now being encountered.

Finally the concepts of mobile launch platforms for clustered rocket engine systems and the possibilities for nuclear propulsion bring the historical development to the close of the current decade. Nuclear propulsion systems would add still another dimension to the thermal protection system even if they are not employed in the terrestial launch engine.

III. REFRACTORY MATERIALS AND THEIR PROPERTIES

A. Commercial Refractory Materials

Commercial refractory materials are supplied in many engineering forms. For this reason the classification system (see Appendix) adopted for this section has been subdivided into: (1) refractory shapes (including refractory brick); (2) refractory monoliths; (3) refractory jointings; (4) granular refractory products. Each of these categories might be divided further into material categories.

Division into material categories is feasible to some extent in the refractory shape class. Hopefully the composition expressed in terms of elemental composition or phase type would dictate characteristic properties and these properties would relate to the utility of the refractory. But this is true to varying degrees because properties may depend also on: (1) the mode of

preparation of raw materials; (2) the fabrication process used; (3) the heat treatment employed; (4) the chemical bond used if the refractory is of the unfired type; (5) the solid phase distribution and type; (6) the distribution of pores (porosity and permeability); (7) the method of placement in service; (8) operational variables in service. Section IV of the report will elaborate on the role of the process variables.

Existing methods of designation may use any or a combination of these as a basis. Committee C-8 on Refractories of the American Society for Testing and Materials has, over a period of some 50 years, evolved type classifications for refractories. They are continually modifying these classes to provide for new materials and new applications for old materials. There is also currently a move toward international standardization.

ASTM Designation C27, Standard Classification for Fireclay and High-Alumina Brick recognizes five different classes of fireclay brick: super duty, high duty, semi-silica, medium duty and low duty. The super duty and high duty classes are further subdivided into three types (depending on intended application) as regular, spall resistant, and slag resistant. The high duty and super duty classes are first differentiated on the basis of their PCE (ASTM Designation C24 Standard Method of Test for Pyrometric Cone Equivalent of Refractory Materials), a test which is essentially a softening point determination using a sample of the pulverized and screened material to prepare a "cone". The deformation end point is compared with a series of standard "cones" which have numbered deformation end points. When heated at a

standard rate in a neutral or air atmosphere these numbered end points correspond to deformation temperatures. Thus, super duty fireclay refractory must have a PCE of 33 (about 3170°F) and high duty fireclay, 31 1/2 (about 3090°F).

The spall resistant types are those which seem to best fill the requirement for the pad surface as related in the previous section. ASTM Designation C38 Standard Method for Basic Procedure in Panel Spalling Test for Refractory Brick relates to the general procedures for determining the spalling loss of a brick panel when subjected to a cyclic heating and cooling treatment. This basic procedure is modified for the type of refractory (ASTM C180 for Fireclay Plastic; C107 for High Duty Fireclay; C122 for Super Duty Fireclay).

Semicilica refractories are a specialized class which have a requirement based on a hot load test in compression and a minimum silica content. PCE is not designated because the pulverized sample does not characterize the deformation of the bulk refractory.

In the high alumina class the correlation between alumina content as determined by chemical analysis and PCE is good enough to use these as a basis for classification. Thus 50, 60, 70, and 80 percent alumina refractories with PCE's of 34 (3200°F), 35 (3245°F), 36 (3280°F) and 37 (3310°F) respectively are recognized. More recently 85, 90, and 99 percent alumina refractory classes have been added.

A more recent (1964) trend to recognize the role of the phase content of a refractory is exemplified by a new classification

of refractories in which the material must be predominantly mullite ($3Al_2O_3.2SiO_2$) with a allowable alumina content limit between 56-79 percent (ASTM C467, Standard Classification of Mullite Refractories). Development of this class indicates the importance of not only designating chemical composition but also characterizing the phase constituents. The need for the classification arises from service correlations which show high alumina refractories with mullite as the primary phase to be different from those in which Al_2O_3 is the primary phase.

The basic refractory group is a typical example of the importance of adapting the refractory to service needs. As a group they are based on formulations utilizing magnesium oxide and chrome ore. The source of the MgO may be mineral carbonates or hydroxides which have been processed to produce an aggregate high in MgO. The MgO may also come from the sea water process which utilizes dolomite ($CaCO_3.MgCO_3$) and sea water or deep well brines to produce MgO referred to as periclase. Most of the high purity periclases (98% + MgO) used in basic refractories come from this source. The final refractory shape product may be either fired or chemically bonded. The shape may also include a steel plate externally applied or internally held. This becomes a part of the steel structure of the furnace in which the refractory is used.

The evolution of the steel industry in which basic slags are employed and the more recent use of oxygen processes have produced a multiplicity of basic refractories for current uses. Developments in the glass and cement industry have also

contributed to product prolification. Classifications, test methods and evaluation are changing alomst daily as these products appear. This group of refractories is one in which mineral composition of raw materials employed or phase constituents of the final refractory is the basis for classification. Products varying from almost 100 percent chrome ore to very high purity periclase are available.

Worthy of note is the fact that at least one NASA sponsored program is in the process of developing high purity periclase as a heat exchange medium for heat exchangers used in high temperature wind tunnel systems.

Insulating refractories as a class are a highly developed product group. They include the insulating fire brick as classified by ASTM C155, Standard Classification of Insulating Fire Brick. The correlation between bulk density and thermal conductivity for these materials is very good so that maximum bulk densities and a temperature limitation based on dimensional change during reheat to a specified temperature may be used as the basis for classification. Thus Group 16 has a temperature limitation of 1550°F (less than 2% change when reheated to this temperature at a specified rate) and a bulk density maximum of 34 pounds per cubic foot, while Group 30 is limited to 2950°F with density less than 68 pounds per cubic foot. Groups 20, 23, 26, and 28 fall in between these end members. As bulk density increases the thermal capacity increases for this group of materials.

Insulating refractories based on high purity alumina and zirconia aggregate prepared in hollow sphere form are also

available to extend the temperature to the 3300°F - 4000°F range of use. Even though these products are more dense than the insulating firebrick group, their thermal conductivities are about the same order of magnitude as Group 30. All of these products have a cold crushing strength not greater than 300 p.s.i. and possibly as low as 40-50. Dense high alumina brick may run as high as 25,000 while fireclay is in the range of 6000-10,000 p.s.i.

Refractory wool products based on high temperature fibers represent another class of materials which, while not load supporting in the normal sense, may have applications because of the variety of textile forms in which they appear (rope, mat, blankets, paper, woven composites, board). With densities in the range of 2 to 10 pounds per cubic foot these materials may have a use temperature to as high as 2300°F. An interesting characteristic of these materials is the fact that the thermal conductivity <u>decreases</u> as the density increases up to the 10 pound per cubic foot range.

<u>Silicon carbide</u> has developed as a commercial material because of its very high thermal conductivity (approximately ten times that of fireclay and high alumina), abrasion resistance associated with its high hardness, and thermal shock resistance. The primary disadvantage has been its poor oxidation resistance when bonded by the conventional fireclay or "silicate" bond. Many products have appeared in which the silicon carbide was essentially an aggregate added to fireclay, high alumina or mullite. The silicon carbide when properly dispersed and bonded in these materials produced increased thermal conductivity and thermal

shock resistance. Silicon carbide is also an example of a granular material made entirely synthetically in an electric furnace process which becomes a premium-priced (compared to fireclay and high alumina) refractory grain for use as a raw material.

To combat the lack of oxidation resistance at least three new products have been in the process of development and are at the evaluation stage: (1) nitride bonded silicon carbide; (2) oxynitride bonded; (3) recrystallized or "self bonded." All of these are attempts to optimize the properties of the material by detailed study of the microstructure and its relation to properties.

Carbon or graphite based refractories have had large scale application in the electrode industry, as mold material for steel casting and as refractory lining for process industry furnaces, particularly the blast furnace. Requirements of nuclear reactors have also generated much work on optimizing the properties of graphite as a reactor core material. It is interesting to note that graphite has high temperature applications with a diversity of requirements: (1) specified electrical resistivity; (2) control of neutron transfer; (3) resistance to molten metals and slags; (4) an ablative material of high enthalpy; (5) material of high strength to weight ratio in temperature ranges to 4000°F. Like silicon carbide, graphite is plagued by its poor oxidation resistance. Again, forms of graphite in the development process are an attempt to optimize widely divergent properties by careful consideration of microstructure.

The development of pyrolytic graphite is an outstanding example of this consideration. Controlled vapor deposition of carbon produces a highly anisotropic, laminated material with a structure more like that of wood than another material class. The properties, therefore, vary not only with respect to the structure but also with respect to the deposition orientation. Properties such as oxidation resistance, thermal conductivity, electrical resistivity, and high temperature strength are sensitive to the orientation so that there may be a directional dependence of one to a hundred orders of magnitude difference.

The multiplicity of products generated by these applications is a matter for separate information retrieval study. The bibliography will reveal this fact.

Materials with commercial significance involving zirconium and its compounds have centered mainly on <u>zircon</u> ($ZrSiO_4$) and zirconium oxide. Zircon is a naturally occuring material with a PCE of at least 42 (3660°F) which is refined to produce a specialized refractory for use in the glass industry, phosphate and sodium silicate processes and in the aluminum industry. It has a unique combination of high density (about 30% greater than fireclay), high PCE and resistance to slags of an acidic nature. On extended use in the range of 2900°F it may decompose to zirconia and silica with an associated breakdown in microstructure.

Zirconium oxide with a fusion temperature of about 4700°F has been limited to some extent by its poor resistance to thermal shock due to a pronounced change in crystal structure from monoclinic to tetragonal in the range of 1800°F. Phase equilibria

studies in systems involving zirconia have produced a product line referred to as "stabilized" zirconia in which the ZrO_2 has been "alloyed" with an oxide (CaO, MgO, Y_2O_3, CeO_2) to produce a cubic solid solution which does not produce significant discontinuities in properties up to its liquidus temperature. The stability of these solid solutions during long time exposure at elevated temperatures is a current subject of research. The high density of ZrO_2 (a 9" x 4 1/2" x 2 1/2" brick would weigh about 16 pounds) would require special consideration in any large installation.

Fusion cast refractories based on the ZrO_2-Al_2O_3-SiO_2 system have shown outstanding erosion and corrosion resistance in the glass industry. The dense fusion cast material resulting from this process has also had abrasion resistant application where extreme thermal shock resistance is not a requirement.

The early use of zirconium oxide was complicated by the fact that it was difficult to obtain HfO_2 free materials. Zirconium oxide has also been a subject for separate information retrieval study as indicated in the bibliography.

<u>Silica refractories</u> might be broadly separated into two main categories: (1) those based on sintered quartzite; and (2) those based on silica glass. The first is the traditional silica refractory which was the mainstay of the steel industry for open hearth roofs prior to the development of basic refractory products. It is still extensively used in glass tank crowns. ASTM C416 Standard Classification for Silica Refractory Brick is intended to cover this product by limiting impurity levels. The contemporary silica refractory of this type is a classic example of the optimum

development of a refractory raw material so that it may be used to within a few degrees of its liquidus temperature (about 3075°F). The phase constitution of the refractory (tridymite, cristobalite, silica glass, calcium silicate) gives it a very low linear expansion in the range above 600°F, but below that temperature structure inversions of the silica polymorphs give it a high linear expansion (3×10^{-6} as compared to 45×10^{-6} inches per inch per °F).

Silica may also be developed as a completely glassy material and handled in the molten state as a glass. The fused, dense material has a coefficient of linear expansion of only 0.30×10^{-6} inches per inch per °F. This combined with low thermal conductivity and relatively high strength makes it unique. The chemical resistance of fused silica is well known to the laboratory chemist. Being a glass it can also be prepared in a variety of forms such as foam, fiber, aggregate, and formed shapes. The aggregate may also be employed in a variety of products such as cements, castables, and thin walled sintered shapes.

Like a glass, it will also devitrify when heated, although the rate of devitrification may be controlled. The product of the devitrification is one which changes the otherwise ideal properties. Development of fused silica materials where moderate refractoriness, dimensional stability, resistance to thermal shock, and low thermal conductivity are required is in process for a wide variety of applications including thermal protection for launch areas.

In closing the section on commercial refractory shapes, mention should be made of the fusion cast refractory process which provides the basis for the production of a material by melting,

nucleation and crystallization from the melt. Melts here involve very high temperatures (circa 5000°F) so the complexities of foundry manipulation of melts may be limiting factors. Products cast in the required shape from the melt now include mullites; ZrO_2-Al_2O_3-SiO_2 types (about one-third to one-half zirconia by weight); chrome-alumina types; high purity aluminas (99 + percent); and sodium modified aluminas (beta alumina). Because of the dynamic conditions under which these melts are cast the materials usually have poor thermal shock resistance due to heterogeniety in composition and structure. Recent developments indicate that this can be controlled by composition and heat treatment adjustments, particularly for small shapes. The outstanding characteristics of this group is its high order erosion and corrosion resistance.

Monoliths are traditionally subdivided into classes based on the method of placement (plastics, ramming mixes, castables, gun mixes). This group could also, of course, be subdivided by material but it is generally true that there are not as many material categories within the various monolith types. For example, plastics are usually fireclay-based, packaged and shipped in prefabricated, unfired, tempered form including the tempering water already in the product. A plastic is ready to be deformed into the shape required at the installation site. It attains the necessary physical properties when subjected to the heat treatment of the furnace operation. Plastics are traditionally raw fireclay-fired aggregate types. (But this does not preclude the appearance on the market of a "plastic" of

another material category.) The "plasticity" of this product group is achieved by the unique rheological properties of the raw clay ingredient when wetted.

A ramming mix on the other hand may be a non-acqeous material, shipped in granular form and placed by pneumatic impact equipment (e.g. air hammer). The composition ranges extend beyond clay based materials. In fact ramming mixes based on magnesium oxide, chrome ore, zirconium oxide and magnesium aluminate are typical examples of commercial materials. Auxillary plasticizers and other additives may be used to produce the requisite properties to aid densification of the material during placement. The material will achieve its properties by the heat treatment resulting from the thermal environment of use and the care during placement.

Castables traditionally have been the refractory equivalent of concrete in which a sized refractory aggregate is mixed with a refractory cement of the calcium aluminate type. When wetted and placed in prepared wooden or steel forms by concreting techniques, the cement hydrates to form a bond in which a "hydraulic setting" mechanism is operative. More recently, however, the appearance of refractories placed in the same manner but having a bond resulting from chemical action other than hydration have appeared. Phosphoric acid, sulfuric acid and chromic acid bonded materials are examples of these types. At the moment the industry is not in complete agreement as to whether or not these should properly be categorized as "castables". As a matter of fact, "chemically bonded" unfired refractories of the basic class or

high alumina category have been available for some time. They utilize bonds of this nature during manufacture of the shape.

Gun mixes are developing also as a broad refractory class covering many composition types. The material for gunning must necessarily be adapted to the equipment used for placement. The refractories engineer must therefore optimize between the characteristics required by the gunning device and the properties required in the finished refractory monolith. The development of the bond begins during the process of gunning and continues after the material is placed. A technique being employed commercially utilizes a dry mix which is transported to the nozzle of the gun under pressure and the bonding ingredients are introduced at the nozzle. This is due to the fact that the bond (for example phosphoric acid or acqueous phosphate complexes) may have a very short working time. Its set is developed almost simultaneously with the impingement of the refractory aggregate mix in the placement site. The properties achieved in the final monolith must obviously warrant the additional care required by this quick set method of placement. Gun mixes may also employ a variety of materials which are essentially concretes utilizing the calcium silicate and/or calcium aluminate type of bond. The curing time on these can be extended to make the gun mix easier to place, but the final properties may not always be as desirable as some of the quick setting materials depending, of course, on the application. Maintenance of a refractory furnace lining by gunning in the hot furnace during or between operational cycles is used in the process industries, particularly in vessels for basic

oxygen process steel. Programmed maintenance by gunning seems to be paying dividends. There may be lessons to be learned here which apply to the embryonic use of refractory monoliths to protect blast deflector structures.

It does not seem feasible, therefore, to list "typical" properties for any of the monolith types. Their successful use will depend entirely on the method of placement and the extent to which the operational variables in the process of use can be adapted to the behavior of materials. There is also no universal monolithic material available which will solve all blast protection problems. The statement is often made that the performance of a material like a castable will ultimately depend on the bond employed and the aggregate becomes essentially a filler. Performance then is a result of the stability of the bond with respect to two fundamental considerations: (1) thermal stability (thermal degradation history) of the bond itself and (2) reaction between the bond and the aggregate at elevated temperatures.

Consideration of examples of some types of calcium aluminate cements is a case in point. Table 2 is an approximation of the PCE and composition of four types of refractory cement in which hydration of calcium aluminate would contribute the bond.

These cements, all of which might appear in any of a variety of castables, not only vary among themselves in refractoriness but might be reactive with an improperly selected aggregate when heated.

TABLE 2

Approximate Compositions of Calcium Aluminate Cements

Type	Total SiO_2 and Fe_2O_3	Al_2O_3-CaO ratio	PCE
I	17.8	1.3	13 (2460°F)
II	8.6	1.3	14 (2550°F)
III	0.4	4.3	34 (3200°F)
IV	6.7	1.3	16 (2720°F)

<u>Jointing</u> is the term coined here to describe a commercial group of materials which are used to join a multiple refractory shape complex which, under ideal conditions, will perform as one structural unit. This involves not only the adhesives and mortars for joining but also the design of the shape, interlocking shapes and anchoring the shape in the structural unit. Many refractory structures which are made of multiple units are constructed with steel jointings. This requires, however, the production of special shapes or anchoring hardware which adds substantially to the cost of the total structure.

<u>Organic based adhesives</u> are being developed for refractory structures. This must be necessarily in cases where the organic is in a temperature gradient zone where its operating temperature is below about 400°F or where the time of exposure is short enough to preclude extensive thermal degradation. Epoxy resins are the most likely group to consider. The chemical term "epoxy" represents a ring containing one oxygen and two

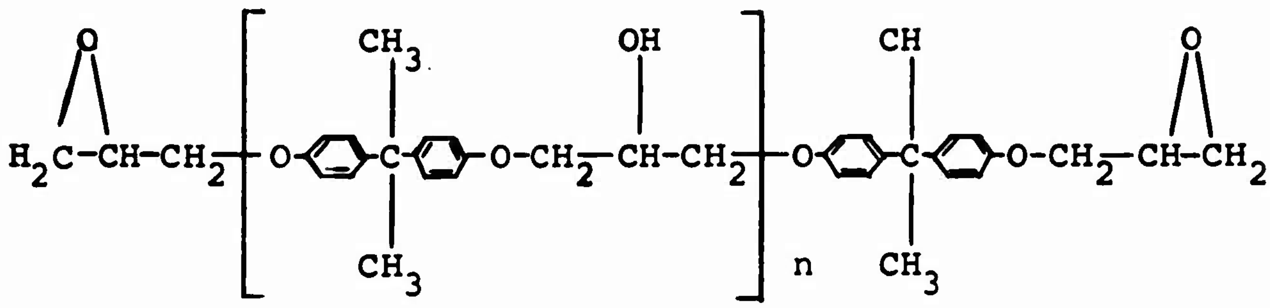
carbon atoms. The resin molecule generally contains more than one epoxy group. The acute angles in the structure produce strain in interatomic chemical bonds, causing the ring to be very reactive.

The three most prominent groups of epoxy resins are diglycidyl ether of bisphenol-A resins, epoxy-novolacs, and cycloaliphatic epoxies. The chemical structure of these resins are shown in Figure 6.

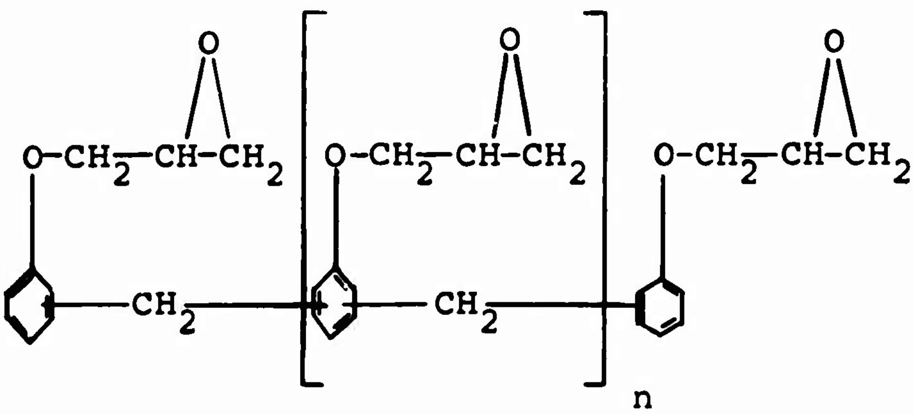
Diglycidyl ether of bisphenol-A epoxy resins are general purpose materials. Their strength properties are considered good as is their hardness, toughness, and rigidity which are retained to 300°F. The greatest asset of these resins is their versatility in processing. They can be used with practically all the epoxy curing agents, and since many of final properties are dependent upon these additions, specific properties can be designed into the final product by the proper curing agent and the amount used.

Epoxy-novolacs have greater heat resistance than the diglycidyl ether of bisphenol-A resins. The structure of these resins is more compact increasing its resistance to chemical attack especially by organic acids. The viscosity is considerably higher making application more difficult. The viscosity can be reduced by additions of standard resins or by heating the resin to 125-130°F.

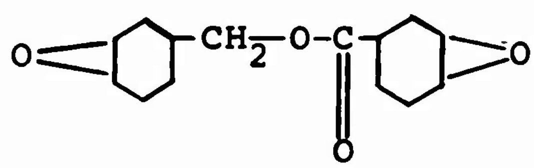
Cycloaliphatic epoxies are noted for their lower viscosity which increases their ease of handling. They have higher heat deflection temperatures than required for curing, and they also have excellent weatherability.



BISPHENOL-A EPICHLOROHYDRIN RESIN



EPOXY-NOVOLAC RESIN



CYCLOALIPHATIC EPOXY RESIN
(CARBOXYLATE TYPE)

FIGURE 6. Structure of Three Basic Epoxy Resins (From Materials in Design Engineering, 1 (61) 111, (1965)

Epoxy novolac and cycloaliphatic both retain their strengths between 400 and 450°F. Of the two, cycloaliphatics are better at elevated temperatures.

The organic adhesive as used in refractory placement, therefore, becomes a combination of a basic resin, hardener and mineral filler. The properties of the adhesive become a compromise of ease of application, high temperature resistance, and weathering resistance. It is interesting to note that the latest ORDL tentative specification for the placement of refractory brick makes reference to a definite Federal specification for the type of resin to be employed (see section V of this report).

Inorganic mortars are divided into classes called heat-setting and air-setting. Heat setting mortars develop their adhesion only at operating temperatures usually in the range of 1800°F to 2500°F. Their composition is usually similar or compatable with the refractory being used. Air setting mortars develop their adhesion during the drying cycle. Chemical bonds (sodium silicate and phosphate types are common) impart the air-setting characteristics to maintain the strength of the joint up to the temperature where the reaction during heat treatment will produce the "ceramic" bond. Here again the mortar is an optimization of factors relating to placement (workability, plasticity, water retention, fineness of grind) and factors relating to utilization (shrinkage during heat treatment, resistance to attack, bond strengths, volume stability).

The purpose of the join in addition to adhesion may be to provide thermal expansion allowance if the refractory structure

is one subjected to repeated heating and cooling cycles. In this case the joint may be expected to be just strong enough to produce an impervious bond but not so reactive as to produce radical changes in the brick structure nor so rigid as to completely confine expansion thrust.

Temperature gradients under which most refractory structures operate may produce a zone of weakness in the range where the chemical bond has begun to deteriorate and the ceramic bond has not fully developed. This is also true of the entire brick structure if it is an unfired chemically bonded material. Anchoring or reinforcing hardware may therefore be an important part of the jointing. In the use of basic refractories for certain regions of the open hearth steel plates actually act as jointings. They become structurally a part of the entire design and thermally a part of the refractory by diffusion reaction at the hot face.

Many abrasion resistant designs also employ a steel cased material, with the joints welded to keep the impingement surface intact during mechanical stress and vibration.

More recently much work has been done on "ceramic adhesives." These are generally of the porcelain enamel variety to which metal powders have been added. The bond developed is probably a result of a diffusion bond in which the adhesive composition is carefully tailored to the properties of the metal to be joined. Ceramic-to-metal, ceramic-to-glass, glass-to-metal, metal-to-metal and ceramic-to-ceramic sealing is an outgrowth of materials technology for electron tubes, transitors, and refractory metal fabrication.

The last group under commercial refractories is granular refractories. There is a large industry built around the production of synthetic refractory grain. The process involves either sintering in rotary or hearth type kilns or electric arc melting and casting of the molten material. The product of sintering is an aggregate much like a cement clinker. The product of arc melting is an ingot. Both of these sources provide a large variety of synthetic minerals all the way from materials like dolomite for furnace bottoms to synthetic carbides for cutting tool use. This industry is developing as a source of synthetic raw materials which may be made available for use in any of the products groups herein discussed.

With the wide variety of product groups and subgroups available, it does not seem that the "handbook approach" to properties is really applicable. The industry is one in which consumer evaluation of tailor made materials and product proliferation are characteristic. Impetuous use of these products exemplifies the trite expression: "A little bit of knowledge is dangerous."

Reference therefore is made specifically to:

> Product Directory of the Refractories Industry of the United States, published by The Refractories Institute, First National Bank Building, Pittsburgh 22, Pennsylvania, 1961.

This is the latest issue of a survey of refractory products listed by manufacturer, plant location, product divisions and brand names. Since 1961, the number of brand names alone has

increased so rapidly that manufacturers are now resorting to coded number systems rather than brand names.

B. Developmental Materials

This section includes refractory materials which have had very limited commercial evaluation to date, or are being considered for possible commercial application in relatively new and less conventional areas. In many cases, raw material and production costs of these materials are at least ten to a hundred-fold that of the commercial refractory materials. These costs could be considerably reduced as the market potential increases.

Table 3 lists property extremes for various refractory ceramics that are typical of this group. Special reference should be made to a specific publication by W. H. Duckworth, et. al. Refractory Ceramics for Aerospace, compiled by Battelle Memorial Institute and published by The American Ceramic Society, Inc., both at Columbus, Ohio. This comprehensive compilation was published in 1964 and was originally administered under the direction of the Ceramics and Graphite Information Center, Materials Information Branch, Materials Applications Division, Air Force Materials Laboratory, at Wright-Patterson Air Force Base, Ohio. It is continually being updated, presently containing over 500 pages of property data.

The oxides have been studied more thoroughly than any other group of refractory ceramics. The oxide group can be subdivided into single oxides (Al_2O_3, BeO, etc.) and mixed oxides (mullite, magnesium aluminates, etc.). Alumina, magnesia, and others with widespread commercial use have been reviewed under the commercial materials section.

TABLE 3. Property Ranges of Some Developmental Refractory Materials [a]

GROUP	MELTING POINT (°F)	DENSITY (gm/cm^3)	THERMAL CONDUCTIVITY 2000°F (Btu)/(hr) (ft)(F)	SUPERIOR THERMAL SHOCK RESISTANCE	% THERMAL EXPANSION at 2000°F
Single Oxides	2710 - 5970 Nb_2O_5 - ThO_2	3.008 - 11.47 BeO - Pu_2O_3	1.2 - 16.0 ZrO_2(stab) - BeO	Al_2O_3 BeO	0.75 - 1.25 Al_2O_3 - VO_2
Mixed Oxides	2700 - 3875 $3CoO.2SiO_2$ - $MgO.Nb_2O_5$	2.60 - 4.58 (b) (c)	1.0 - 310	(d)	0.25 - 1.0 (b) (c)
Carbides	2800 - 7010 $Cr_{23}C_6$ - TaC	2.04 - 17.34 CaC_2 - W_2C	10 - 25 HfC - SiC	SiC	0.5 - 1.5 SiC - UC
Borides	2250 - 5880 Ni_2B - HfB	2.63 - 16.72 MgB_2 - W_2B	12 - 35 Zr_2B - HfB_2	(e)	0.5 - 1.0 ZrB_2 - CrB_2
Nitrides	2730 - 5990 CrN - HfN	2.25 - 15.8 BN - Ta_2N	3 - 12 Si_3N_4 - BN	BN Si_3N_4	0 - 1.0 BN(f) - BN(g)
Silicides	2730 - 4980 $TiSi_2$ - B_3Si	4.15 - 15.58 $TiSi_2$ - V_3Si	10 - 20 $MoSi_2$ - WSi_2	$TaSi_2$ WSi_2 $MoSi_2$	0.75 - 1.5 $MoSi_2$ - USi
Sulfides	2370 - 4040 TaS_2 - ThS	2.68 - 15.58 MgS - US	N O D A T A		
Aluminides	2660 - 3920 TiAl - CrAl	3.90 - 8.38 TiAl - UAl_2	N O D A T A		1.0 - 1.5 CrAl - UAl_2

(a) range lists the material that lies at each end for that particular property; (b) silicides; (c) aluminides; (d) not as good as Al_2O_3 and BeO; (e) conflicting data on thermal shock resistance; (f) perpendicular to pressing direction; (g) parallel to pressing direction

The properties of the single "pure" oxides vary greatly. Alumina and beryllia have good thermal shock properties while magnesia is considered fair at best. The strength of the oxides is generally not considered as good as those of other refractory ceramics, but their excellent oxidation resistance sets them apart from all other groups.

Beryllium oxide is noted for its exceptional thermal conductivity which is considerably greater than any other oxide. A dense sintered beryllia body will conduct heat better than iron and its alloys. By use of calculated thermal stress factors BeO shows better thermal shock resistance than any other oxide until it reaches a maximum resistance at about 600°F. Here the rapid decrease in thermal conductivity with increasing temperature influences such calculations to indicate a drop in thermal shock resistance.

Barium oxide, calcium oxide, and strontium oxide all react readily with water to form their respective hydroxides. They will also hydrate in air, the rate of which is controlled by the density of the fired piece. The melting point of calcia (4690°F) and its abundance (limestone and dolomite) have long made it a desirable refractory material; however, the problem of hydration has limited its use. Barium and strontium oxide are presently not used commercially in large quantities.

Titania (TiO_2) exists as anatase, rutile, or brookite. Rutile is the most stable and common form, but all three modifications can exist at room temperature. The melting point of titania is relatively low compared to other oxides and limited data have been published on its mechanical properties.

Although thorium oxide and uranium dioxide have exceptionally high melting points, density and availability have limited their use except for nuclear applications.

Mixed oxides can be compounds, solid solutions, or composite mixtures. Some of the silicate mixed oxides (Mullite, fosterite, etc.) have had widespread commercial use in various refractory applications. Many of the other mixed oxide systems are better known for their electrical properties. Magnesium aluminate is an example of a mixed oxide which may have commercial significance in the near future. The mixed oxide system involving magnesia, alumina and zirconia is under study currently and may reveal future materials development areas.

The carbide group includes some compounds having the highest known melting points. Their lack of oxidation resistance has limited their use as refractories. Except for silicon carbide, their thermal shock resistance is not always considered good. The group is also known for its hardness. Other than silicon carbide, which has been discussed under commercial refractories, tungsten, titanium and zirconium carbides have been developed for cutting tool applications and for relatively small precision shapes for other uses.

Borides are known for their hardness and chemical stability. They are reported to retain their strength at high temperature and have good oxidation resistance. Their commercial uses are still limited even though extensive investigation of their properties have been undertaken in the last few years for possible applications in the aerospace industries.

The nitrides do not form as many stable high temperature compounds as the borides and carbides. Silicon nitride (Si_3N_4) and boron nitride (BN) are the only two nitrides with any commercial value at the present. Silicon nitride (Si_3N_4) is very resistant to an oxidizing atmosphere. The thermal shock resistance of this material is considered to be very good. Silicon nitrides and silicon-oxynitrides are being studied as bonds for silicon carbide to render that material more generally applicable for refractory applications where oxidation resistance has been a limiting factor.

Boron nitride may be likened to carbon in its properties. The usual form with the hexagonal lattice is soft and strongly anisotropic in its properties. A second cubic form is somewhat analogous to diamond and is a product of extreme pressure-high temperature studies. It is almost as hard as diamond and has possibilities as a high temperature cutting tool since it does not begin to oxidize except at temperatures approaching (3500 + F°).

The silicides have a lower melting point than the carbides and borides; however, they are known for their oxidation resistance and hot strengths. Molybdenum disilicide is know for its exceptional oxidation resistance, due to the formation of a glassy coating which softens at high temperatures and renews itself. Besides molybdenum disilicide, the sidilicides of tungsten and tantalum are considered to have the best possibilities for commercial refractory applications.

The melting points of the aluminides are considerably lower than those materials previously discussed. Only three of

them melt above 3000°F. Some of the higher melting compounds have shown good strength and oxidation resistance to 2000°F. Detailed research studies of these and similar intermetallic compounds are being conducted.

The physical and chemical stability of the sulphides has discouraged property studies except in the case where special environments admit their use.

Thus the developmental group contains some rather exotic materials as well as familiar ones. Refractory technology will progress when some of these proceed to the production stage. This will not happen unless close correlation with service evaluation is maintained.

C. Refractory Materials Systems Based on Ablative Materials and Refractory Coatings

Two interesting basic concepts of thermal containment arise from aerospace vehicle applications for refractories where the heat flux levels and temperature are too high to expect a static solution to the thermal containment problem: the ablative system and the radiative system. In the ablative system, heat flux is accommodated and dissipated by a mass-transfer heat absorption in a vaporizing medium. If the thermal accommodation expected is ΔH and the rate of mass loss during the process is $\dot{m}$, then the effectiveness of the system depends on $\Delta H/\dot{m}$ being a very large value and occurring over an acceptable time period. The expendable thermal shield therefore, partially destroys itself in the process of performing its function. This requires detailed knowledge of the vaporization enthalpy of refractories at extreme

temperatures. Alumina, beryllia, magnesia, and organic polymers of large hydrogen content become outstanding materials when contained in a refractory matrix which maintains its structural integrity due to the cooling effect during ablation. Transpiration or micro-transpiration in tungsten composities is a typical example of this operational system.

Ablative systems capitalize on the high heats of vaporization, sublimation, and dissociation of materials to provide thermal protection to a structure employing four mechanism: (1) the intrinsic thermal capacity of the system; (2) the heat energy absorbed during physical changes such as vaporization, melting or sublimation, and during chemical changes such as depolymerization; (3) the heat energy absorbed or "blocked" by the transpiration cooling effect of the gaseous products as they pass through the outer layer of the material, through the boundary layer, and into the atmosphere; (4) energy dissipated by thermal radiation.

The first two factors predominate at lower heat fluxes while the last two become prominent at extreme heat fluxes which would normally produce temperatures much above the melting points of any known materials.

Table 4 lists the heat absorbed by materials from 300-5000°K obtained by integrating their specific heats. This fact is again demonstrated by Table 5 which lists the effective heat of ablation of various materials and composites. Graphite is outstanding in this group.

Ablative materials can be grouped as: (1) organics which decompose into a gas; (2) materials which sublime and

TABLE 4

Substance	Heat Absorbed, Cal./gm.
H_2 gas	67,000
$(CH_2)_n$ organic plastic	24,000
$(CH)_n$ organic plastic	20,600
C graphite	16,670
$(C_6H_{12}O_6)_n$	6,760
BeO beryllia	7,080
MgO magnesia	5,500
SiO_2 silica	2,800
Be	9,876
Cu	1,600
$(C_2F_4)_n$ Teflon	6,300
H_2O	14,500
He	3,525

TABLE 5

Material	Effective Heat of Ablation (Btu/lb.)
Copper	397
Aluminum	544
Stainless Steel	433
Molybdenum	1,045
Zircon	1,110
Mullite	1,225
Alumina	1,570
Linen Bakelite	16,800
Nylon	7,850
Graphite	52,000
Teflon	3,130
Phenolic-Nylon	4,480
Phenolic-Refrasil	7,530
Phenolic-Glass	9,600

and react with air; (3) materials which melt then vaporize; and (4) composites which pyrolyze and char. Teflon is an example of the first group. Its effective heat of ablation is high and it decomposes around 700°F. Graphite is an example of a solid that sublimes. After sublimation the carbon atoms react with dissociated oxygen atoms to form carbon oxides. Glasses are in the category of materials which melt and vaporize. Fused silica with its good thermal shock resistance is an excellent example. After melting, the silica forms gaseous oxide species (monoxide or dioxide), but the melt has a viscosity high enough to maintain a stable surface configuration in the liquid state.

Composites become the real candidate refractory materials systems to utilize the ablative mechanism of thermal protection. Often one component of the composite is in fibrous form, although many theoretically feasible systems incorporating organics, ceramics and metals are in development stages.

The radiative system assumes a heat exchange by absorption into a heat sink and subsequent radiation emittance to the atmosphere. Here, of course, a knowledge of the temperature and wavelength dependency of emittance, absorptance and reflectance are the characterization parameters for the refractory system. In a dynamic situation of this nature, the thermal diffusivity is an important thermal property. Thermal diffusivity is physically the thermal conductivity divided by the product of specific heat and density. This parameter arises from the treatment of the transport mechanism of heat as a diffusion process.

The radiation system is an ideal place for the utilization of coatings. These may take the form of thermal control utilizing a knowledge of their optical properties, because the temperatures of intended use are high enough to extrapolate data on optical properties to the entire thermal spectrum. They may also take the form of insulating, thermophototropic, oxidation resistant, impingement protective, or ablative coatings.

Special reference should be made to J. D. Plunkett's recent survey: NASA Contributions to the Technology of Inorganic Coatings, NASA SP-501 Technology Utilization Division, National Aeronautics and Space Administration, Washington, D. C., prepared under contract to NASA by Denver Research Institute, Denver, Colorado, 1964.

IV. EFFECT OF PROCESS VARIABLES ON CRITICAL MATERIAL CHARACTERISTICS

A. Characterization of Refractory Materials

In contemporary refractories engineering the term "characterization" refers to the ability to quantitatively describe an engineering material at three levels of understanding: (1) the atomistic level (angstrom units); (2) the microstructural level (microns); (3) the property or phenomenological level.

The atomistic level relates not only to the identification of elements present, but also to the spatial arrangement of their sites in a three-dimensional lattice. It is this

arrangement of atoms to which modern x-ray diffractometry addresses itself. The probability that this idealized lattice is repeated infinitely in three dimensions without distortion is, of course, very small. In real materials, therefore, defects in the form of (1) impurity atoms; (2) lattice defects of form or distortion; and (3) lattice defects of omission of atoms become the rule rather than the exception. The practice of precalcining refractory materials to produce an aggregate ingredient also leads to the formation of partially developed structures of a defective nature. This may actually be desirable in producing a well bonded refractory in the final product form.

If the single crystal with a perfect lattice (a condition practically impossible to attain) is considered as one end member of a structural series, the polyphase, polycrystalline material with its defects, pores and impurity phases might be considered the other end member. It is the function of <u>microstructural</u> characterization therefore to place the material at its proper level in this structural series.

When the modes of fabrication available for the production of a <u>competitive</u> refractory material are considered and when the wide range of properties required is tabulated, it is realized that a fully dense material is neither probable nor desirable except in very special cases. The metallurgically ideal microstructure consisting of a fine-grained, fully dense material is based on the assumption that mechanical properties alon dictate the utility. In refractory technology <u>control</u> of

porosity at some optimized level is the actual basis for producing real materials with resistance to thermal spalling; desired thermal conductivity; and required densities.

It is also known that raw materials can be beneficiated only to a degree which is compatible with the economics of production and utilization of a refractory. Therefore, impurity phases may be present simply because they come with the major desired phase as auxillary phases which were not economically feasible to remove. In the early days of refractory technology this situation was so prevalent that the actual microstructure was an accidental result rather than an objective. In the production of unfired refractories, the prepared components of a refractory are combined in a manner which will allow them to react with their environment of use to produce the stable microstructure. Microstructure in this instance only becomes a matter of record after extensive service evaluation and post mortem study. The microstructure may have been optimized entirely on the basis of reaction in service with only minimum attention to mechanical properties.

The complete characterization of the microstructure of a refractory should therefore include: (1) quantity, size, shape and distribution of all crystalline phases present including a representation of continuity of critical phases; (2) quantity, size, shape and distribution of pore phase, both closed micropores and grain boundary porosity; (3) the amount and distribution of glassy phases present; (4) the chemical composition of all phases present including a representation of compositional gradients in each phase.

To place the property or phenomenological level of understanding in its proper perspective, the progress of characterization of a refractory material must be considered. Figure 7 shows this progression diagramatically. It starts with a particulate material which must be characterized as to: (1) size distribution and shape; (2) surface area per unit weight and density; (3) crystallography or petrography; (4) chemical activity (e.g. hydration behavior); (5) degree of chemical purity or controlled impurity.

A combination of differential thermal analysis, thermogravimetric analysis and shrinkage behavior may be used to further define the response to thermal treatment. This is particularly true if a prereacted aggregate is prepared.

By proper blending with liquid media and processing additives which vary in function from being binders, lubricants, flocculents, plasticizers or deflocculents to being fungicides or bacteriacides for aging stability, a batch is produced. The only economical recourse to characterization at this point may be purely simulative in nature. Therefore, trial batches may be fabricated and fired as a real expedient approach to a decision as to the acceptability of the batch. The characterization of the response of the batch to forming is beyond the scope of this report. It must be said, however, that this is an area of propriety and also of much needed development work. Fabricating large complex shapes is particularly troublesome.

The melt forming processes may offer some degree of simplification in batching. But the advantage gained at this stage

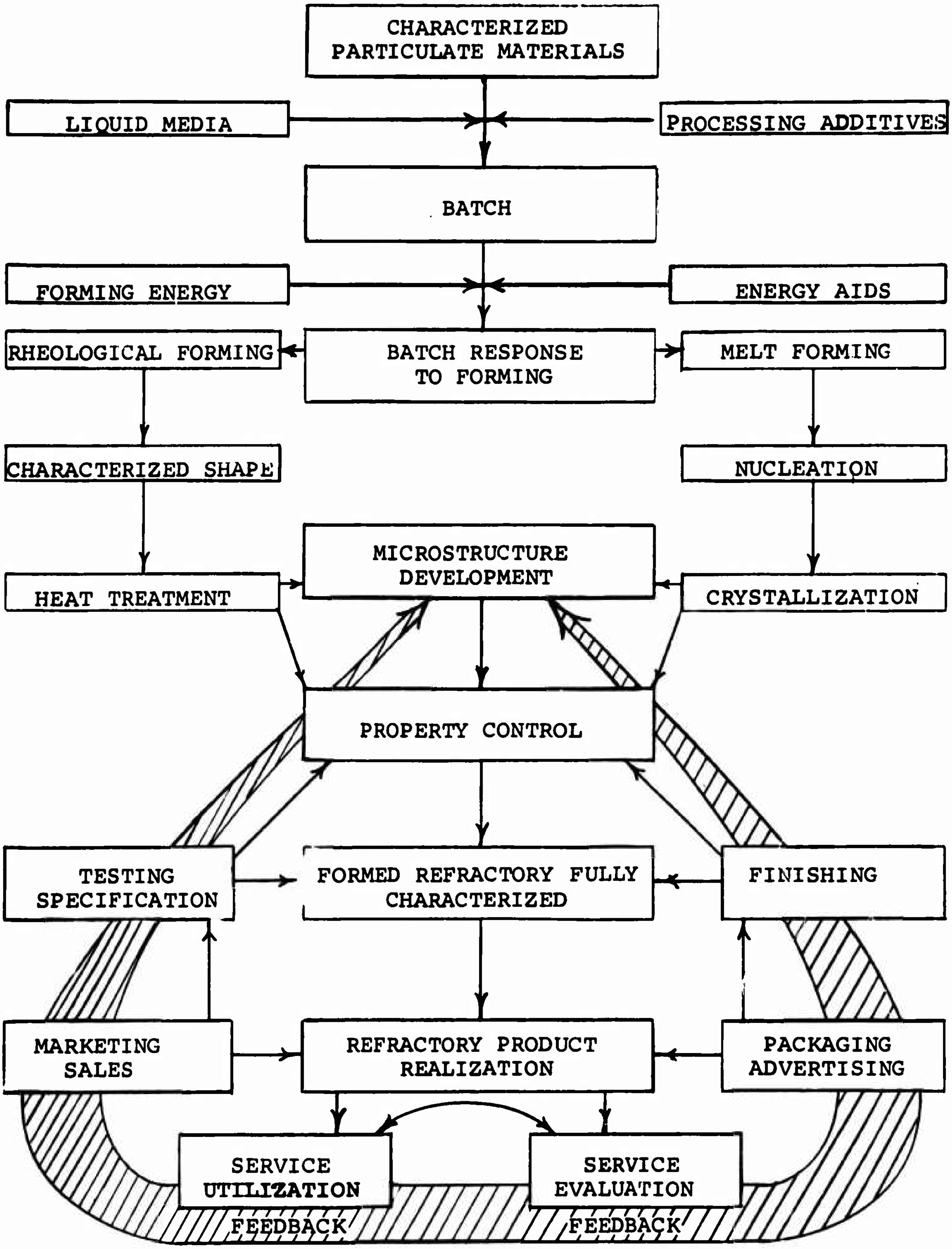


FIGURE 7. Progression of Characterization of a Refractory Product

may be lost by the complexities of the foundry manipulation of very high temperature melts in a process such as the fusion casting operation. Melt forming includes the controlled nucleation and crystallization of glasses ("Pyroceram") and plasma and flame spraying techniques. It also includes the production of refractory glasses like fused silica.

Rheological forming refers, of course, to the traditional fabrication of liquid media-solid systems by slip casting, plastic forming, pressure forming, isostatic pressing, ramming and the multitude of variations thereof.

Finally, by a process of heat treatment, sintering, or the control of crystallization of a melt a microstructure is developed. The property control which is possible is a direct result of this microstructure. After required finishing operations and a testing evaluation to check specifications, the formed refractory, fully characterized, is now realized. The finishing operation must not be passed lightly because this may be one of the most costly operations when close dimensional tolerances are required.

The refractory shape can still not be characterized as a product until its properties, intended use and availability have been advertised and it has been put in a package satisfying asthetic and customer requirements. Its final application must also be the detailed consideration of a technical marketing and sales approach.

The utilization of the refractory product with its associated evaluation in the service environment provides the real

basis for the information feedback shown in figure 7. The technical cooperation between suppliers and consumers at this stage is a necessary condition of operation. This joint action is perhaps more typical of refractory use than any other field of materials engineering.

This feedback closes the loop and makes the improvement of refractories a continuous process. Note that the loop originates at the evaluation-utilization level of characterization and after influencing marketing, sales, testing, specification, packaging, advertising, and finishing it converges on property control and microstructure development to start around again. Thus, refractories are literally tailor made for their application in a specific materials system.

An overall characterization of refractories is therefore based on performance in service in combination with the more detailed analytical methods available to the laboratory and pilot plant.

B. Critical Properties for Blast Resistant Materials

The behavioral categories considered to be important with respect to blast resistance are primarily thermal shock, erosion resistance, and reactivity. The bibliography card system has been based on this subdivision. Each of these are behavioral considerations quite complex within themselves and individually dependant on composition, microstructure, fabrication, heat treatment and operational variables.

A considerable volume of literature has been published on thermal stress, thermal shock, and thermal spalling. Thermal stress results from gradients introduced by differential thermal expansion strain. Thermal shock is often evaluated by determining the degree of deterioration of a mechanical property parameter like strength or elastic modulus. Thermal spalling refers to that degree of thermal shock failure which results in loss of the material due to fragmentation. As a matter of fact, the ASTM test referred to previously uses a loss in weight as the basis for differentiation of fireclay brick. Other test methods specify the evaluation of the degree of mechanical deterioration by determination of elastic or strength moduli before and after (or during) repeated thermal shock cycles. This brings up the point that thermal stress may be "absorbed" within a refractory without sufficient damage to produce complete failure. But in absorbing the energy of thermal shock the material may undergo limited permanent physical damage at the microstructural level leading to less significant bulk property changes. Thermal shock may be likened to impact loading wherein the origin of the stress in thermally induced rather than pressure induced.

The stress distribution arising from thermal expansion is a function of: (1) intrinsic properties of the material; (2) geometry of the shape; (3) type of heat transfer within the refractory; (4) type of heat transfer to the refractory.

The first and second of these normally considers the refractory from a continuum mechanics viewpoint leading to the calculation of "thermal shock resistant factors." In analyzing

these structures, a common result is a resistance factor, R:

$$R = \frac{S(1-\mu)}{E\alpha}$$

where S is a strength parameter, μ is Poisson's ratio, E is an elastic modulus parameter and α is the coefficient of thermal expansion (assuming it to be linear). This factor must be modified first by consideration of geometry so

$$R' = \frac{S(1-\mu)}{E\alpha} \times \text{ shape factor.}$$

In many situations, however, the geometry is fixed so there is little or no improvement possible by modifying shape.

Secondly, it must be modified to consider the degree of thermal impact loading. The surface stress resulting from such loading is a function of:

$$\frac{h}{k} \times \text{ shape factor}$$

where h is the boundary-dependant heat transfer coefficient and k is the material-dependant heat transfer coefficient within the refractory. (Since the latter is often so closely related to thermal conductivity, it might be assumed to be equal to the thermal conductivity.) If h is very much larger than k, the thermal conductivity of the material has little to do with the thermal shock resistance because a surface stress will be confined to a limited thickness producing an extreme gradient condition. However, if k is of the same order of magnitude as h, the conductivity may play an important role in minimizing the gradient effects. The first case may be treated as a completely dynamic one in which thermal diffusivity is the controlling thermal property. The second case approaches the steady state where thermal conductivity is the controlling thermal property.

Of course, both cases still depend on the original materials parameter R so that a resilient material with high strength to elastic modulus ratio or one with very low thermal expansion might also endure high order thermal impact loading with minimum damage. The thermal shock resistance of a carbide or beryllium oxide might be good because the high thermal conductivity is the controlling factor. But the thermal shock resistance of silica glass may also be good because the exceptionally low expansion coefficient and low thermal diffusivity are controlling factors.

These considerations result from assumptions that elasticity exists in the temperature range encountered and that the material is homogeneous and isotropic. A second approach to understanding thermal shock resistance arises from consideration of the microstructural level of relief of thermal strain.

For catastrophic failure to occur <u>propogation</u> of the fracture as well as nucleation of the fracture must be considered. This propogation requires that the strain energy released during fracture must be equal to or greater than the surface energy of renewed surfaces. In polyphase, heterogeneous, brittle materials the strain energy in a unit volume of material may be considerably <u>less</u> than surface energy at grain boundaries. Refractory technologists know that proper aggregate sizing in the material introduces heterogeniety which will interrupt thermal shock crack propogation. Actually heterogeniety in the form of pores, plastically deformable constituents or fibers have all been employed to produce thermally resilient composites.

As temperature increases, plastic yield or even viscous flow changes the condition from one of true elastic stress to one of relaxed stress gradients. Here the stress gradient at the heated surface is not so steep or perhaps non-existant. The viscous or plastic heated layer may act as a boundary. This "pyroplastic" condition may sustain large pressure induced stresses and at the same time be markedly resistant to thermal shock. This is often exhibited by silica-rich materials which, when heated above their softening point, still maintain stability by virtue of the very high viscosity of the surface liquid backed up by a zone of the cooler material which has structural stability.

The refractories engineer also recognizes a condition called "structural spalling" which develops in refractories subjected to temperature gradients. This condition of a refractory which develops under the influence of temperature, pressure, atmosphere, and corrosion agent gradients during service leads to property discontinuities by diffusion-generated metasomatism. The result is a thermally unstable zoning of the refractory. This has been observed not only in furnace refractories but in nuclear fuel elements, rocket nozzles, and space vehicle thermal shields. In aerospace applications a multicomponent refractory system selected to match a known gradient condition has been employed to minimize this condition. This is also practiced in conventional furnace design.

The following conclusions may be made with regard to minimizing thermal shock failures:

(1) Resistance to thermal shock in the elastic state is favored by high strength to elasticity ratio.

(2) The proper role of heat transfer must be assessed to carefully defined environmental as well as material parameters.

(3) Prestressing or conversely, designs which afford thermal stress relief by mechanical means (all parts in compression, numerous small parts confined in compression) should not be overlooked.

(4) Release of thermal strain energy in a heterogeneous composite may cause microstructural changes but may scatter thermal shock energy to maintain bulk structural integrity.

(5) A viscous boundary layer created on the surface of a heated refractory offers a means of relieving stress by modifying surface gradients.

(6) Otherwise controlling boundary layer (water cooling, vapor sweeping) conditions may reduce thermal stress buildup.

(7) A material system with low thermal conductivity and low expansion coefficient may confine gradients to a very thin boundary.

(8) A material of high thermal diffusivity may reduce gradient effects where surface heat loadings are not severe.

(9) Control of radiative heat transfer in thermally transparent materials may be a means of controlling thermal stress buildup (perhaps by utilizing coatings or infra red absorbing additives).

Erosion resistance relates primarily to operational conditions for refractory materials. Erosion with respect to movement of molten slags and glasses and high speed particle stream impingement has been studied in the process industries. Invariably, the work is related only to a special set of conditions which are applicable in a specific area. Most of the work has been based on abrasion testing so that actually a distinction between abrasion and erosion is not always clear in the test program. The refractories committee of ASTM, through a subgroup of committee C-8, has been studying a test for the abrasion resistance of refractories utilizing a sand blasting apparatus.

Even the definition of erosion varies depending on the application area. For the purposes of the launch facility, the term "erosion" must refer to mechanical abrasion by solids suspended in a moving, high temperature, impinging fluid producing loss of material by wear. This would mean that: (1) the velocity of movement of the stream; (2) the characterization of particles in the fluid; (3) the temperature of the fluid; and (4) the angle of impingement must be the critical variables. The dynamic characterization of the moving fluid often goes beyond defining velocity and temperature because turbulence originating near the source of the stream or in the impingement area of the stream might develop cavitation. The accumulation of the wear products and particles from the stream at high temperature may therefore produce corrosion. Corrosion in this sense implies deterioration by chemical attack in combination with loss of material by wear.

Studies of the material aspects of reducing wear in this erosion-corrosion complex have led to the following conclusions: (1) shear strength is of great importance; (2) surface roughness (as exhibited by porous materials) and unevenness or projections from the surface promotes wear; (3) surface spalling increases the rate of erosion by roughening the surface; (4) material hardness is a single intrinsic property relating to wear resistnace. Some work has actually been done in "hot hardness" testing to establish temperature dependancy of this latter property. Work with refractory materials in linings for chutes to handle hot moving ore or dust streams has supported the above statements. Invariably the dense, hard material, properly surfaced and properly mounted to avoid mechanical distress during vibration produces the optimum design. Paradoxically this type of material usually has poor thermal shock resistance. Since the strength of a refractory decreases as the porosity increases and the erosion resistance decreases as the porosity increases a correlation between strength and erosion resistance is to be expected.

Because the refractory material is often based on a bonded aggregate, the erosion resistance is only as good as the bond employed. Refractory shapes with bond developed by firing normally exhibit optimum erosion resistance characteristic of the intrinsic properties of the material. But when a hard, abrasion resistant aggregate is bonded chemically, such as in the case of a castable, the deterioration of the bond is the weak link which reduces erosion resistance. Fused cast refractories have been quite successful in abrasion resistant applications when they

are free of casting defects and have high surface density. The justification of the cost of these materials must always be balanced against the cost of periodically replacing a less expensive but less abrasion resistant material.

The special type of erosion encountered in blast deflection has shown that the angle of impingement is one of the most important factors in minimizing erosion. The SPREE (Solid Propellant Rocket Exhaust Effects) program being conducted by Martin of Denver for NASA, Marshall Space Flight Center at Huntsville, Alabama is an example of an erosion study found in this survey. Based on both cold jet and hot jet test firings, the program is establishing detailed criteria for design of deflector configurations to turn the exhaust plume and reduce the heat loading and impingement damage. It is also significant that "cheap, trowelable concrete-type refractory materials" may be candidate materials because of the probably lower cost and ease of maintenance. Turning the plume with a precisely designed deflector may minimize the direct impingement damage, but does not eliminate the problem of handling the exhaust gases and their heat radiation to other portions of the facility. This work is covered in more detail in Section V (Materials Utilization and Testing) of this report.

Earlier success with steel deflector configurations of the "Mexican hat" or "morning glory" type had also established the importance of geometrical configuration of the deflector on erosion resistance. Jet engine blast fences which are currently operational also verify the same concept.

The third material characteristic which must be considered for launch facility refractories is that of chemical reactivity

or chemical stability of the refractory. Even for short duration exposure: (1) stability of the refractory itself; (2) stability in contact with other refractories in the system; (3) stability of jointing materials; (4) stability in contact with combustion products of the engine and (5) stability in contact with fuels are all important.

In liquid fueled engines the combustion gases may be either oxidizing or reducing, dependant on the fuel-oxidizer ratio, although oxidizing conditions would probably prevail. Reducing conditions cannot be eliminated from the discussion particularly with solid fueled engines. At present, the reactivity may be less important because of short exposure times. Factors (1) and (2) above then, may be of lesser importance than (3), (4), and (5). The rate of reaction becomes the important consideration. The rate of reaction of slags with refractories is exponentially related to temperature. Very often an increase of a few degrees will change the rate of reaction by a factor of two or more. The condition of operation in launch facilities is also one in which any reaction products formed would be carried away. This increases the potential for reaction because of the absence of the formation of a protective reaction layer.

In refractories of the castable type, it is interesting to examine the stability of the calcium aluminate bond from a viewpoint solely related to the effect of temperature. Table 6 is a representation of the changes in a calcium aluminate cement (after Schneider, Journal American Ceramic Society, Vol. 42 (4) 1959, p. 190) with temperature, assuming calcium monoaluminate to

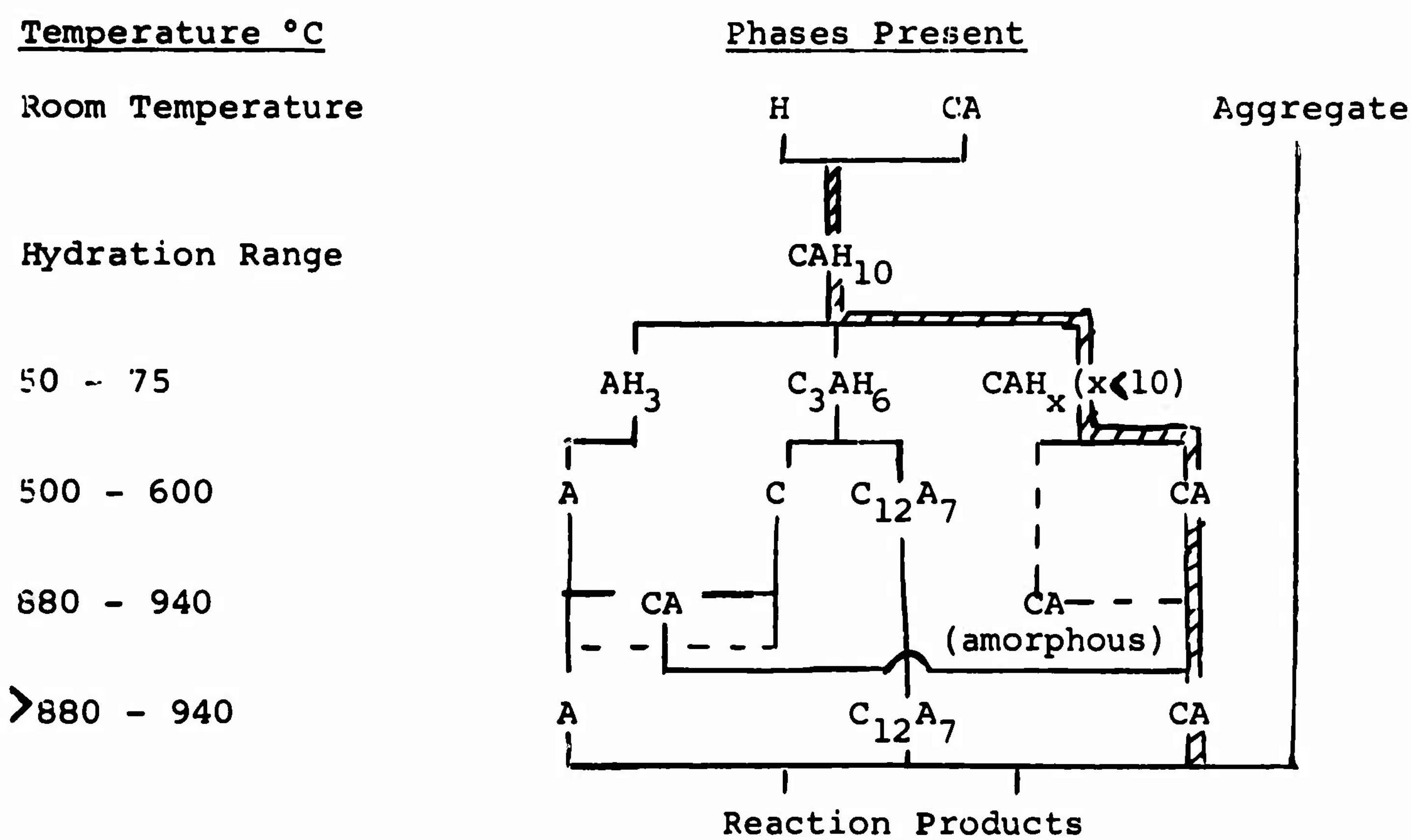
be the only original compound in the cement. After the original hydration reactions to produce the "hydraulic set", changes continue to occur in the cement phase due to the breakdown of the hydrated phases at elevated temperatures. At a temperature as low as 940°C (1725°F) reaction between the dehydrated cement and the aggregate may occur. Note also that in the range of 500 - 600°C (1025°F) the cement has completely dehydrated. Mechanical properties and bonding strength have begun to deteriorate rapidly even below this temperature. The structural integrity will have to be maintained by the cooler section of the concrete monolith or reinforcement hardware will have to be provided to support the weakened zone. This will have to be hardware thermally and chemically stable in the environment of use.

V. MATERIALS UTILIZATION

A survey of materials utilization becomes the most difficult part of a compilation of this type because of the widely separated locations of facilities. Early in the program it was decided that a site visitation to Kennedy Space Center would yield the most information in the time available. (This University of Illinois survey is a one man-year program with a one man-month extension.)

Reports resulting from a preliminary survey questionairre did not yield much in the nature of actual materials utilization experience. It did, however,yield extensive data relative to the nature of materials testing at simulative scaled down

TABLE 6



$H = H_2O$; $A = Al_2O_3$; $C = CaO$; the double line shows the major dehydration path, although all reactions may occur depending on the nature of the calcium aluminate compounds in the beginning cement.

facilities, product information, and theoretical studies of blast effects in the missile field, petrophysics and soil mechanics. Quite a number of reports were concerned with recommendations for future utilization of refractory materials.

This section, therefore, is not to be considered as comprehensive but rather as exemplary of problems encountered. Even the visit to Kennedy Space Center was not completely comprehensive.

Materials utilization is readily subdivided into: (1) materials testing, (2) materials specification; and (3) materials application. Whether specifications appear prior to application or after may be a matter of the particular utilization area. The normal course of events would presume service experience prior to formulating complete specifications, but the rapidly evolving space program may not always allow the accumulation of the type of complete service records to which refractory engineers are normally accustomed.

Figure 2 shows an ORDL test facility designed originally to test materials for runways. Its major features are the miniature rocket assembly and the rotating sample table. Movement of aircraft over a paving surface is simulated by the rotating table. The miniature rocket can be repositioned to provide several passes over the same material. This facility proved to be applicable to materials testing for launching pad use.

Figure 5 shows the SPREE simulated launch site to which previous reference has been made. This work is based on the implementation of a programmed investigation in which Phase I was

a study to investigate data available from industrial and governmental sources. This data was used to develop model systems to predict behavior for solid motor exhaust environments. Phase II was divided into two parts. Part A utilized cold jet streams and hot jet streams from single nozzle configurations to verify theories associated with model system performance together with various configurations of deflectors coated with several types of protective coatings. Part B is to validate essentially the same elements for multiple exhaust nozzles. A consideration of protective techniques for components of the launch facility other than the deflector is also to be a part of this program. It recognizes the problem of correlating model studies with full scale firings. It also emphasizes the importance of following through to the examination of launch sites <u>after</u> firing to provide feedback based on service utilization.

It should be emphasized that a test facility of this program simulates the movement of the rocket away from the deflector surface. The rate of this movement has a very profound effect on total erosion of the deflectors.

The characterization of the rocket exhaust was primarily a matter of determining the size and temperature of alumina particle streams and radiometer studies to measure heat radiation from the exhaust plume. Since the radiating particles in the plume reached a temperature of near 4000°F, the heat radiation problem could be a major one. Radiation from an isothermal plume can be predicted from the classical Stefan-Boltzman equation where the heat flux, q is related to plume temperature,

T, by:

$$q = \varepsilon \sigma T^4$$

where ε is the plume emissivity and σ is the Stefan-Boltzman constant. Their work pointed out that the alumina particle stream influenced the emissivity by

$$\varepsilon = 1-e^{-A}$$

where

$$A = \frac{1.5\ ML}{Vpd}$$

in which M is the particle mass flow rate, L, is the radiation path length, V is the particle velocity, p is particle density and d is particle diameter. Variation in the plume temperature distribution both radially and along the path of travel made this calculation cumbersome. Actual radiation measurements in the vicinity of a plume need to be made.

Contrary to expectations the aluminum content of the fuel used (14 to 22%) did not influence erosion rates as much as expected in the case where curved deflectors were employed. Chamber pressure was a more significant variable, but the mass flow rates apparently were too variable to draw any definitive correlations.

The cold jet portion of the program reiterated the importance of deflector geometry. By use of a Coanda-type deflector (see Figure 18), the pressure of an impinging jet stream could be reduced as much as 75 percent and the plume effectively turned. Figure 18 shows a possible evolution of deflector configuration as the severity of blast effects increases. These still require dissipation of heat after the blast has been turned

from the impingement zone.

One materials recommendation concluded that fused silica tile (Masrock) could be used on flat plate deflectors and epoxy-polyamide (Martite FS) was suitable for ablative protection of surfaces parallel to the axis of motor exhaust. It is interesting to note that silicon carbide with oxynitride bond, (Crystolon 63) and silicon carbide with silicon nitride bond (Crystalon N) exhibited low erosion using a small scale motor (nozzle throat diameter 0.360 inches). In a Titan III subscale (1000 lb. thrust) material evaluation a specimen drive assembly to simulate actual launch conditions by moving the refractory away from the rocket exhaust, fused silica was considered to be the optimum protection material for horizontal surfaces subjected to high impingement angles. Silicone rubber was found to be suitable for joints. These recommendations were made after testing a wide variety of materials.

Another example of a materials oriented program was the work conducted by The Marquardt Corporation under contract AF 04 (611)-4304 entitled Uncooled Refractory Lined Flame Deflector. This work was necessitated by the requirements of the Edwards Air Force Base test facility (page 13-16 of this report) in which emphasis could not be placed on water cooling because of the complexities of pumping systems required in areas where large quantities of water were not readily available. The major facility here was a static test site expected to be versatile in duration of flame-on time (10 to 300 seconds), impingement gas temperatures up to 5000°F, velocities of Mach one, engine thrusts from 5000 to

to 400,000 pounds and a wide variety of liquid fuels producing corrosive, oxidizing and/or neutral environments (such as LOX/RP-1, N_2O_4/hydrazine, hydrogen/fluorine, pentaborane/hydrazine). Early (1960) approaches to employ a deflector for ejection cooling air utilizing the energy of the exhaust to drive the ejection system showed that providing quantities of air to significantly cool the exhaust plume was not practical.

Eighty-eight refractory materials were procurred for use in an evaluation program which included a most comprehensive survey of commercial refractories plus many developmental materials including: aluminas; alumina cement-emery; beryllia; at least twelve castables, including some with special silicon carbide (self bonded and all types of bonded SiC known at that date); fused silica; zircons and zirconias. Composites included zirconia and refractory metals (Mo, Zr, W); magnesia and refractory metals (Mo and W); phenolic impregnated alumina, zirconia and magnesia; tar bonded dolomite; zircon plus graphite; magnesia plus graphite and eleven phenolic impregnated fiber glass materials.

The evaluation was conducted in several phases. The physical property phase included thermal expansion, compressive strength, hot modulus of rupture, density, and porosity. The next phase ("thermal drop" test) exposed the materials to an oxy-acetylene torch. This was scaled up successively to a three inch ramjet burner and a six inch sudden expansion burner (JP-4 and RP-1 fuel). All of these simulated extreme temperature conditions. An additional hydrogen torch provided hydrogen-fluorine testing at higher temperatures. Using the six inch

burner, full scale segments of "J" deflector configuration were used. These were well instrumented with thermocouples, optical pyrometer and a Shaw two color pyrometer (to measure exhaust temperatures). The full size "J" sections were stainless steel covered with anchored castable on which were bonded refractory brick sections. The impingement zone was located so it did not strike a joint directly.

Conclusions from this test program cautioned again that laboratory tests could not be expected to translate results to full scale operation. The deflector could be considered as being divided into two zones: (1) a flame containment region and (2) an impingement zone. The most important properties were thermal shock, corrosion and chemical reaction resistance in combination with "resistance to high temperature", the latter presumably meaning simply high melting point. Nitride bonded silicon carbide was concluded to be the best material for the impingement region but it was realized that there might be variations in the same material from different manufacturers. Density, thermal conductivity and specific heat were recognized as being the most important intrinsic properties.

A later report (July, 1961) scaled this laboratory study up to include a deflector design study for 5,000 and 30,000 pound thrust horizontal fired rockets; a 40,000 pound vertical thrust rocket; a mobile thrust stand and a deflector combined with a hot stack. The design developed the open "J" with a flame impingement angle of 30° and turned through 90° by a curved section. The impingement refractory could be replaced readily, the

size of the replacement zone being dependant on the thrust capacity of the engine. Nine inch straights were the normally recommended shape backed by insulating castable and maintaining 1/16" maximum mortar joints. If a number of deflectors were to be built, the recommendations allowed for total fabrication by the manufacturer (conforming to contour requirements) in which tongue and groove edges would eliminate mortar joints. Alumina castable, nitride bonded silicon carbide and phosphate bonded silicon carbide mortar were material recommendations. The mortar recommendation was the result of a study of mortars, both commercial and experimental, in which the chemical attack by hydrogen-fluorine gases was of major concern.

The final report (August, 1961) of this work concluded that: (1) dry deflectors using ablative or erodable materials function as well as water-cooled buckets; (2) for short runs, castables or other air setting monoliths were a good optimization because of cost considerations; (3) for longer, higher thrust conditions, a combination of a dense, high thermal capacity, high thermal conductivity material and an ablative material should be used; (4) impingement areas require the dense material; (5) other areas require thermal insulating materials; (6) the dense material should be covered by a thin layer of ablative material to protect it from initial thermal shock during the initial impingement.

The report concluded with specifications for furnishing, delivery and installation of refractory materials for the static missile test site at Edwards Air Force Base, California (dated October, 1960).

The most recently dated specification found in this survey is the Revision of ORDL Tentative Specification issued September, 1958, and revised in draft form January, 1965. The 1958 issue was prepared to assist several Engineer Districts and engineering groups concerned with plans, engineering and installation of launch facilities. It was essentially the one followed in the construction of Kennedy Space Center complexes like the Saturn complexes 34 and 37. It has been submitted to the Chief of Engineers, U. S. Army Engineers, for publication as a Guide Specification. Although it is not practical to publish it in detail in this report, it should be emphasized that it recognizes the problems of placement of pad structures exclusive of blast deflectors and as such also is cognizant of the fact that after the flame has been turned by the deflector, refractory problems still remain in dissipating the hot gases. It is entitled, Guide Specifications for Military Construction, Pavement, Refractory Brick for Critical Areas of Aircraft and Missile Facilities. As emphasized earlier in this report, it covers generally the super duty fireclay brick class (ASTM C27) with added requirements with respect to blast resistance, placement, size variation, freezing and thawing and jointing and surfacing.

A new round of development of refractory concretes was also evident in the survey. There are materials which would be classified perhaps as "ablative monoliths." Their use would be where emphasis on economy of fabrication and maintenance must be stressed. They are not necessarily a new breed with respect to composition, but rather with respect to the concept of their

use. Rocketdyne report E-2036 entitled, Refractory Concrete Flame Deflector Development Study, December, 1959, was a study of concrete mix design and testing of fabricated slabs in the exhaust flame of a LOX/RP-1, 1,000 pound engine with 45° impingement.

This work was initiated due to the failure of a static test facility (up to 150,000 pound thrust) utilizing steel plates or portland cement concrete deflectors. Attempts to use a high alumina cement with emery aggregate in this major facility had produced a variety of unreliable results due to variability of the aggregate.

The refractory concrete mix design experiments showed that by properly selecting the cement and aggregates and by controlling the normal parameters of cement-aggregate ratio, size distribution of aggregate (particularly fines) and cement-water ratio, several concretes applicable to deflector fabrication could be produced. Table 7 is a summary approximation of four types of concrete which may have possibilities. This table is based on the Rocketdyne work, but also on a general knowledge of the refractory field, since the characterization of refractory concrete constituents is not always a non-proprietary subject. The base material was generally an insulating castable of low thermal diffusivity (volcanic origin). The others may be applied as toppings which are "ablative" to some extent but also react to form a fused surface which has a tendency to stay in place as a high viscosity liquid which cools to form a glass.

TABLE 7

	Base Concrete	High Temperature Resistant	Thermal Shock Resistant	Neutron Field
Cement* Type	I or II	III	III	III
Possible aggregate type	Lightweight, cellular moderate PCE	$ZrSiO_4$	$ZrSiO_4$, SiC, two aggregates	SiC, graphite, two aggregates
Cement - aggregate ratio (approx.)	0.37	0.33-0.42	0.50-0.63	0.47
Cement - water ratio (approx.)	2.1	2.3	2.3	2.3

* See Table 2, page 35 for approximate characterization of these types of cements.

These concretes carry the thermal degradation of the cement well into the reaction range shown in Table 6. The reaction product is perhaps an oxide of high temperature stability formed by the reaction of the cement and aggregate. This oxide is dispersed in an ablative, glassy, high silica liquid as the reaction proceeds. The Rocketdyne report emphasizes that the source of this reaction would be primarily the aggregate fines and the cement, hence the importance of distribution of fines and the ratio of cement to fines (recommended about 2.4). It is important also to minimize or completely eliminate sources of carbonate in the aggregate. The reaction must come from the calcium aluminate and zircon fines only. Although zircon seems to be the aggregate implied here, it is possible that other alumino-silicates might yield similar results.

Correspondence accompanying this survey indicates that concretes of the base type in Table 7 were specified for launching

silos at Vandenberg Air Force Base and for surfacing Los Angeles International Airport jet aircraft runways. The extent of application of the material, however, has not been indicated.

Application of these castables to areas other than the primary deflector zones, such as in the entire pad area, may not be economically feasible considering the cost of anchoring hardware, placement and higher cost of the topping concretes. At least one cost analysis has shown, for example, that for an installation like Saturn complex 37, refractory brick installation costs would be only about 35% that of special concretes of this type.

Studies of the erosion of refractory concrete for deflector protection have shown a marked decrease in erosion as distance from the nozzle increases. If R is the erosion rate, and D is the location of the impingement zone (region of highest erosion on the deflector) then approximately:

$$R = Ae^{-BD}$$

D is usually expressed in terms of nozzle diameters and A and B are constants depending on the installation and material. Increasing the distance between deflector and nozzle is one approach; but, of course, this would produce added problems, not the least of which is plume thermal radiation exposure.

To gain experience in the <u>application</u> area of materials utilization, a field trip was made to Kennedy Space Center January 26-28, 1965.

The early Redstone - Jupiter launch facilities sites 5 and 6 used a steel "Mexican Hat" type flame deflector and

castable refractory block pad. Regular concrete surrounded the castable section and was also used for the base of the pad. Launching a missile from this type of arrangement resulted in erosion of the castable refractory around the metal deflector. The affected area also had a glassy phase present on the surface where the flame temperatures were the highest. It was noted that the plain concrete around the castable section was attacked by the hot gases from the missile. Attempts to measure the te[illegible] on this pad by embedding thermocouples did not produce entirely satisfactory results because of the rapid loss of transducer mountings during a firing.

A conference with A. P. Green Firebrick Company at Mexico, Missouri was held May 25, 1965. The full extent of their work in the application of castables and super duty fireclay brick was discussed. Their experiments to simulate the temperature rise in insulated panels of refractory castable utilized an adaption of the ASTM Panel Spalling Test equipment. These experiments were the only data found relating to thermal diffusion in both wet and dry heated castable panels. The heat source (furnace at 2500°F) may not have simulated the exact heat flux of a launch condition, but they did indicate temperature stability of pad configurations including the expected temperature rise at interfacial zones. They also helped provide information relating to anchoring hardware, possible interlocking brick joints and placement procedures.

An interesting experiment showed that the color of a reheated castable sample was characteristic of the reheat temperature. Color panels were made available to show this

correlation. Although the color may also be due, of course, to other variables, it helps visualize exposure conditions in the field for at least one type of castable.

Complex 12 used an all-steel flame bucket which contained rows of small nozzles for water transpiration cooling during launch. Although the system performed satisfactorily, the operational cost apparently was excessive because of the amount of fresh water required.

Complex 36a used for launching the Atlas - Centaur missile utilized a concrete flame bucket deflector and concrete trough convered with six inches of calcium aluminate cement bonded castable to channel the exhaust flame. Cavitation damage caused by flame impingement was extensive. Formation of a quantity of glassy material on the surface had occurred. Because large quantities of water drained through the troughs during and between launchings, the monolithic material had shown severe thermal spalling. Attempts at patching the walls and floor with ordinary concrete had been ineffective. Figure 8 shows the damage in this flame bucket. Figure 9 shows extreme damage in the runoff trough and under the gantry track.

Two of the hydrogen burn-off ponds were inspected at Saturn sites. At one of the ponds it was observed that the concrete above the water-line had badly deteriorated due to the high temperature of the burning hydrogen. The other pond had been repaired with a capping of calcium aluminate castable containing a fireclay aggregate. This capping was used just above the water line of the pond. Since the pond had not yet been used, there was no indication of the performance of the capping.

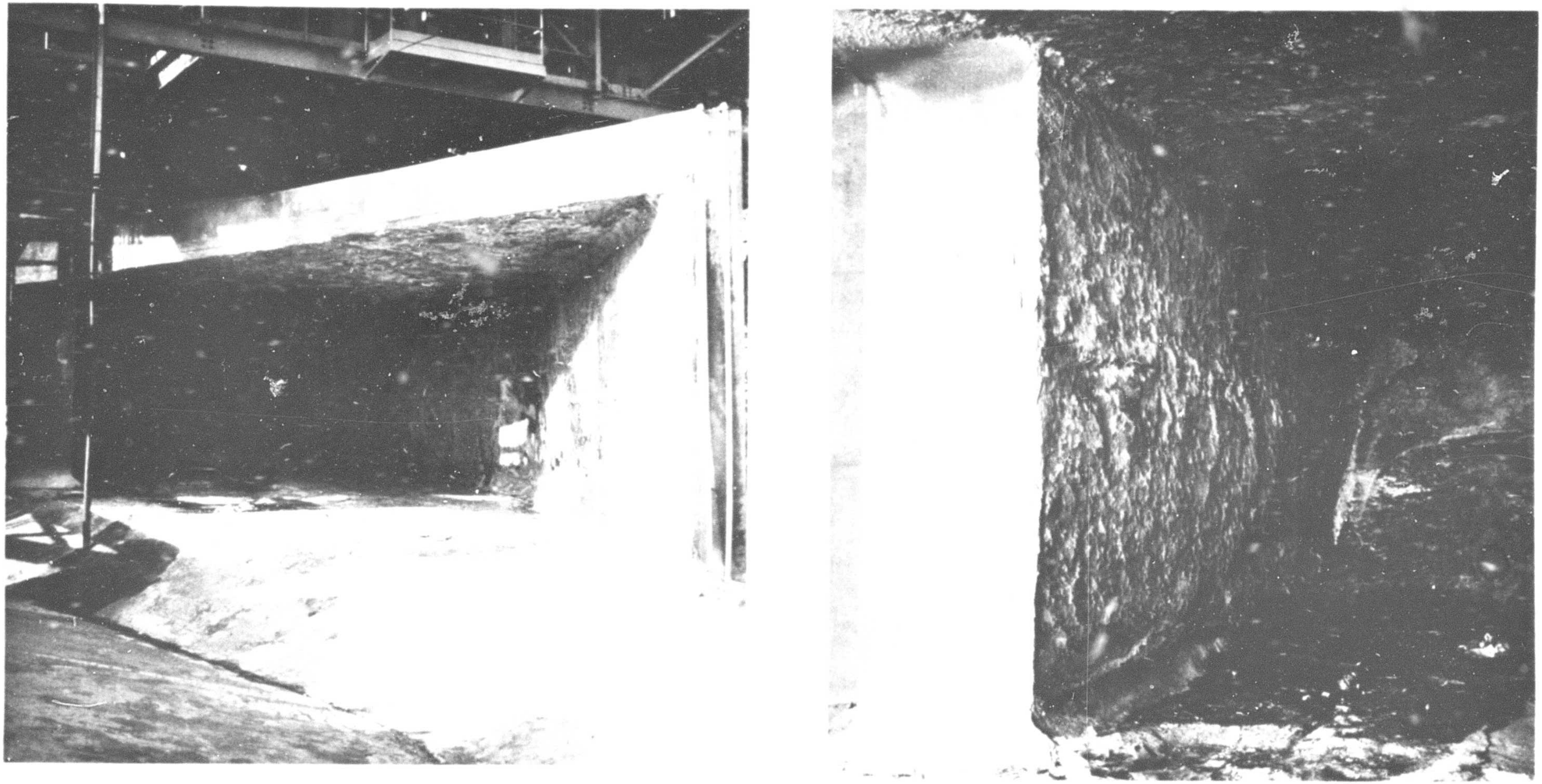


FIGURE 8. Refractory Concrete Flame Bucket Complex 36A KSC Showing Extensive Spalling Due to Thermal Degradation. Right Photo Shows Closeup of Vertical Wall.

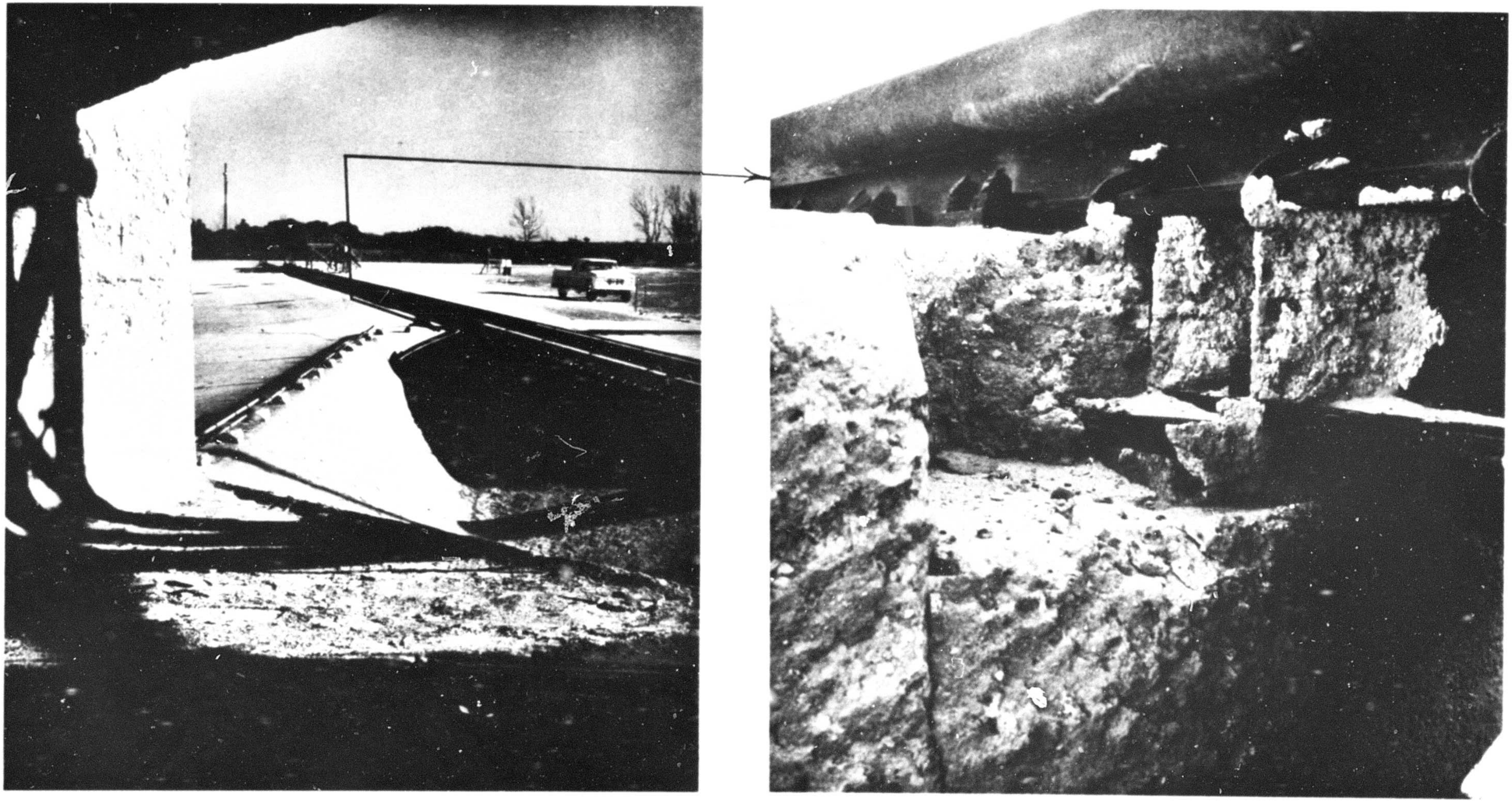


FIGURE 9. View of Cooling Water Runoff Trough Showing Concrete Thermal Spalling. Closeup of Damage Under Gantry Track Shown on Right. Complex 36A KSC.

This installation is an emergency facility in that if the rocket cannot be fired, the hydrogen is drained off to burn in this water cooled area. Figures 10 and 11 show two views of the concrete configuration in this installation.

At the Saturn launch sites, the "Mexican Hat" deflector used in the early facilities has been replaced by a massive knife-edged deflector made of steel. This deflector weighs about 150 tons and is 22 feet high. The curvature of this deflector knife edge is critical. This design is the result of extensive studies of geometric configuration and its effect on erosion resistance.

Figure 12 shows a general view of launch complex 34 to help establish a concept of the size relation of the paving area to the missile launch support structure. Careful inspection of this photograph will reveal the cooling water ducts ending in nozzles which spray cool the pad by flushing water across the area immediately adjacent to the location of the deflector. Cooling water is also provided in a ring surrounding the support just below the rocket nozzle.

The insert in Figure 12 shows the importance of extreme care in masonry practice. This brick joint has been damaged by vehicular traffic probably because of slightly irregilar joint matching. The refractories engineer knows, of course, that these bricks were not designed as paving bricks; but nevertheless, vehicular movement over the pad prior to launch is a necessary condition of operation. Perhaps thought should be given to a temporary cover sheeting in critical areas.

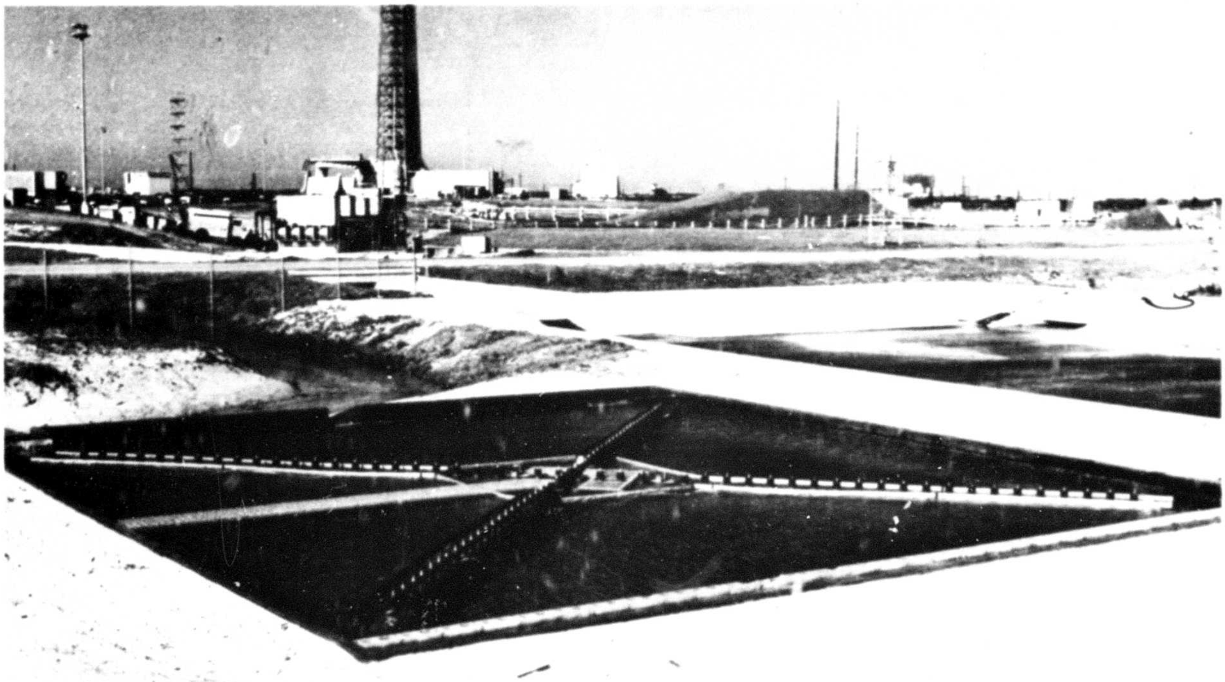


FIGURE 10. Hydrogen Burnoff Pond at Complex 37 KSC

FIGURE 11. Closeup of Repaired Wall and Wier Section. Calcium Aluminate Bonded Refractory Concrete Used to Repair Top Section.

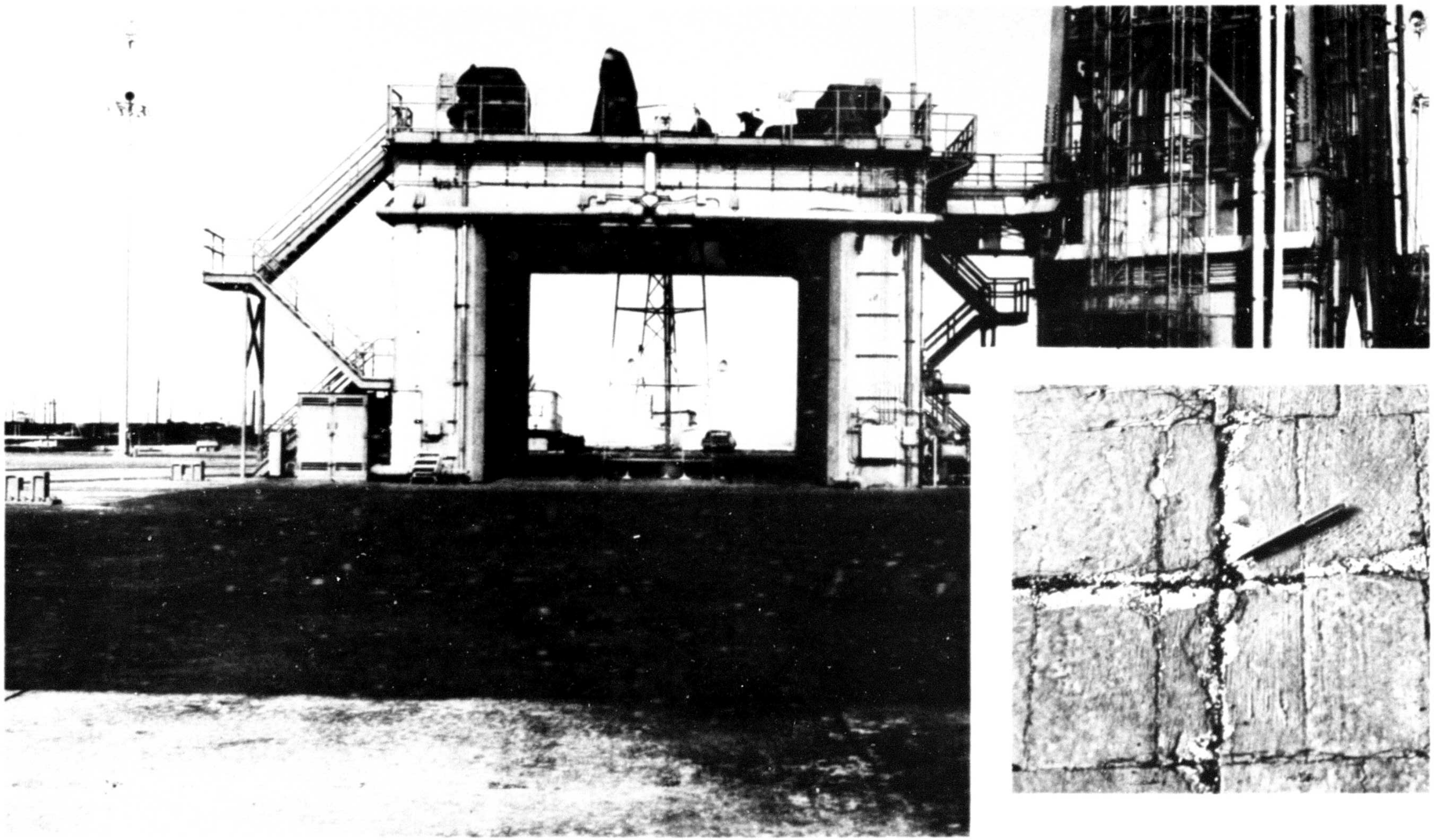


FIGURE 12. Launch Complex 34 KSC. Grey Area is Super Duty Fireclay Brick Pad Bonded to Base with Epoxy Adhesive. Surface Smoothed with Calcium Aluminate Cement Grout. Insert Shows Brick Joint Damage Due to Vehicular Traffic.

Figure 13 shows the installation of a pad complex (complex 34) in progress. Super duty fireclay refractory brick have replaced castable and gunned materials on the pad. The brick are "butt-jointed" and held in place by epoxy adhesives. A coating of fine calcium aluminate cement grout is applied to seal the joints between the brick and produce a monolithic surface. The effectiveness of the system is dependent upon the care in material placement. An exposed brick edge will break the laminar gas flow creating a turbulence that causes cavitation pockets which can tear out the placed brick. Even the placement of the brick with brand up is thought to be undesireable because it interrupts the smooth surfacing. This was also quite important where rail traffic had moved over the embedded tracks installed in the pad. Again studies of proper masonry placement procedures may have improved performances. Shapes other than nine inch straights may be needed in some critical areas. In fact, the entire subjects of the proper shape to use is a matter for consideration.

Figure 14 shows a steel flame deflector without refractory monolith surfacing. These deflectors are moved by rail to a position beneath the rocket exhaust. Figure 15 shows a deflector in launch position and with monolith coating applied. This "coating" is of the type referred to in Table 7. It is not known whether topping compounds had been applied to the base material ("Fondu Fyre").

Figure 16 shows a mobile launch vehicle to which fused silica tile have been applied to protect the steel structure.

FIGURE 13. Installation of Refractory Brick Pad Complex 34 KSC. Note Epoxy Adhesive Layer Between Base Pavement and Brick Courses. (Courtesy A. P. Green Fire Brick Co.)

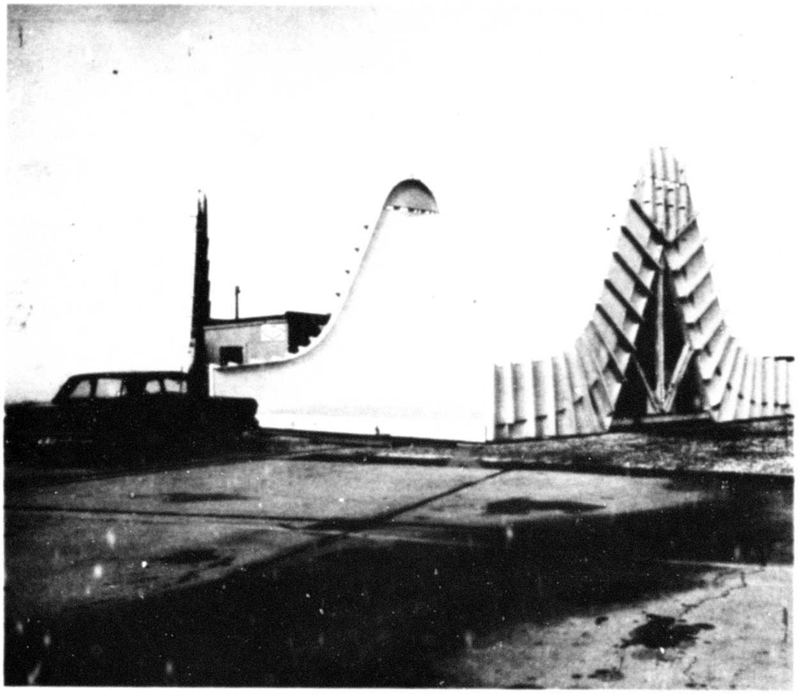


FIGURE 14. Steel Flame Deflector Without Refractory Coating Complex 34 KSC.

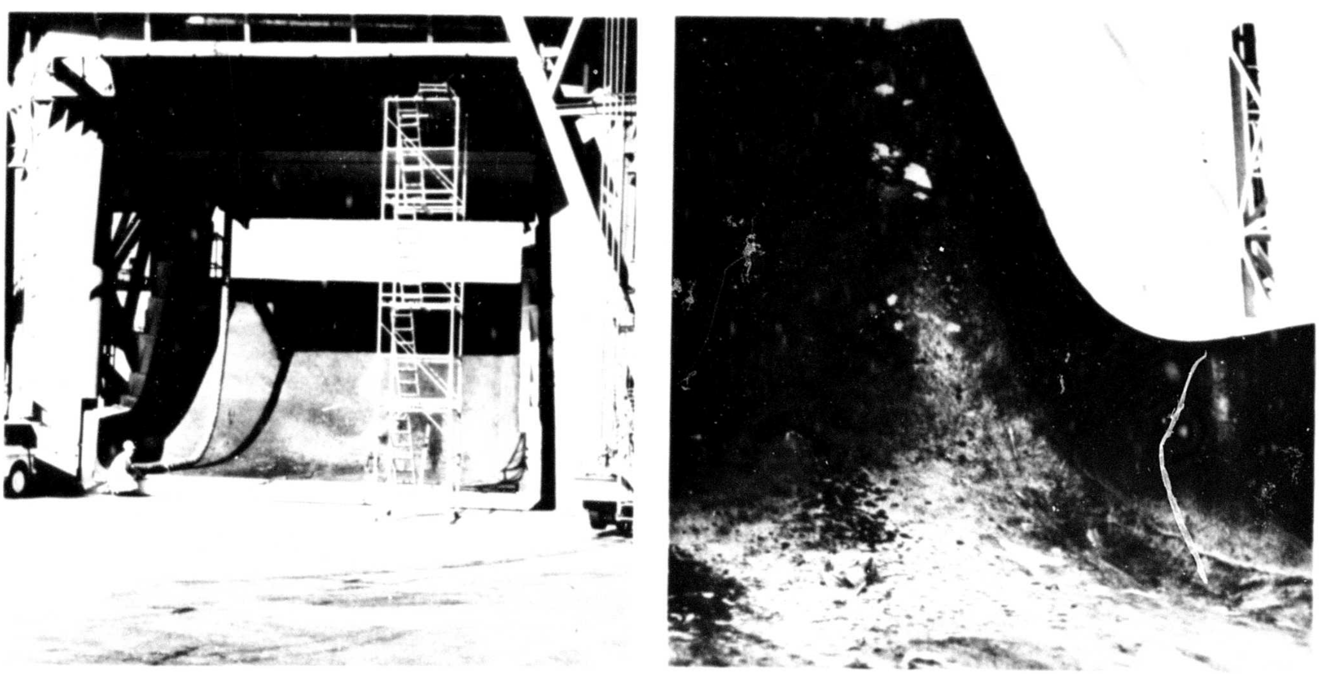


FIGURE 15. Flame Deflector in Launch Position with Refractory Coating Applied. Right Photo Closeup of Refractory Coating Near Impingement Area. Complex 37 KSC.

FIGURE 16. Mobile Launch Vehicle for Titan III-C Showing Fused Silica Protective Tile. (Courtesy Glasrock Products, Incorporated.)

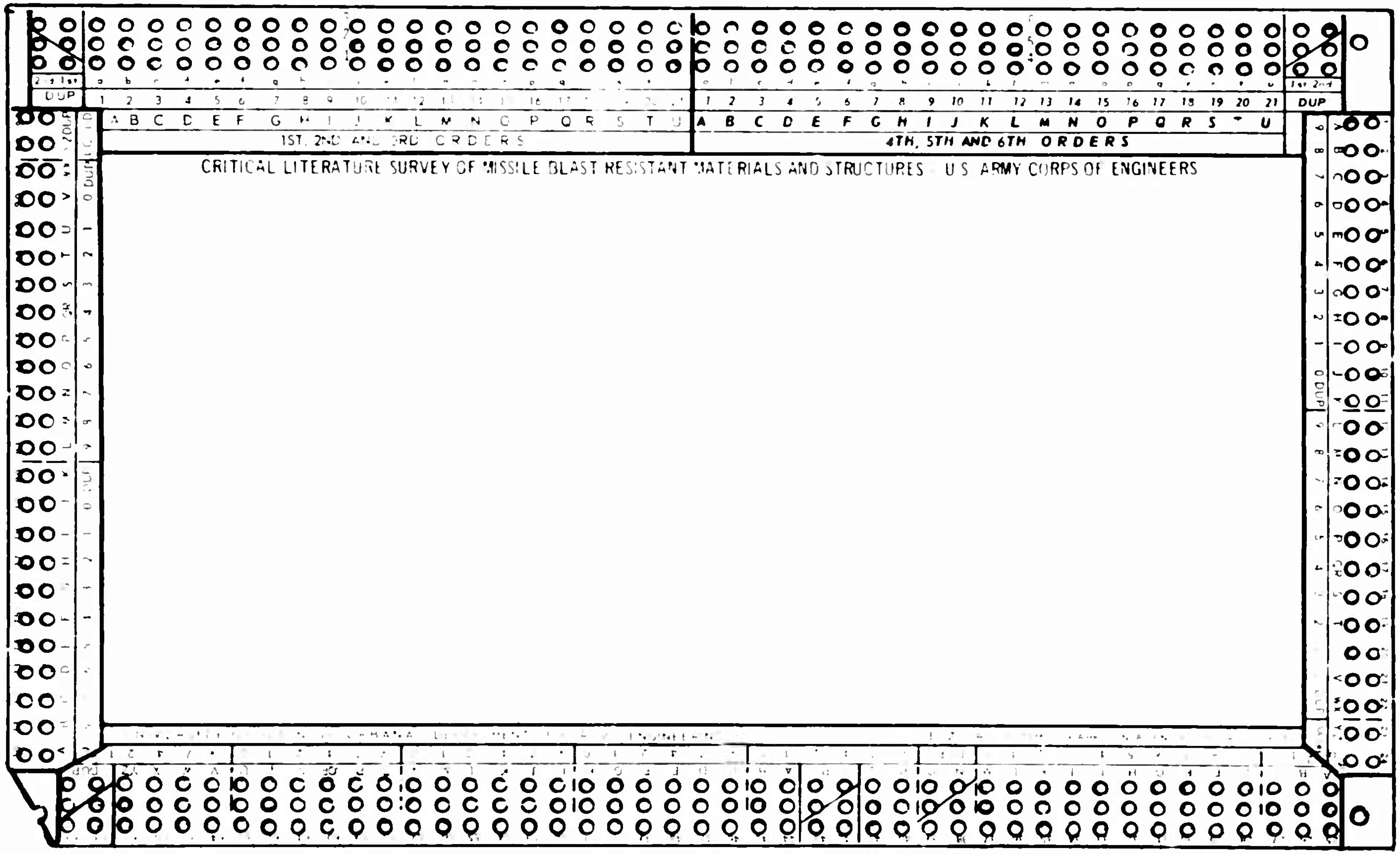
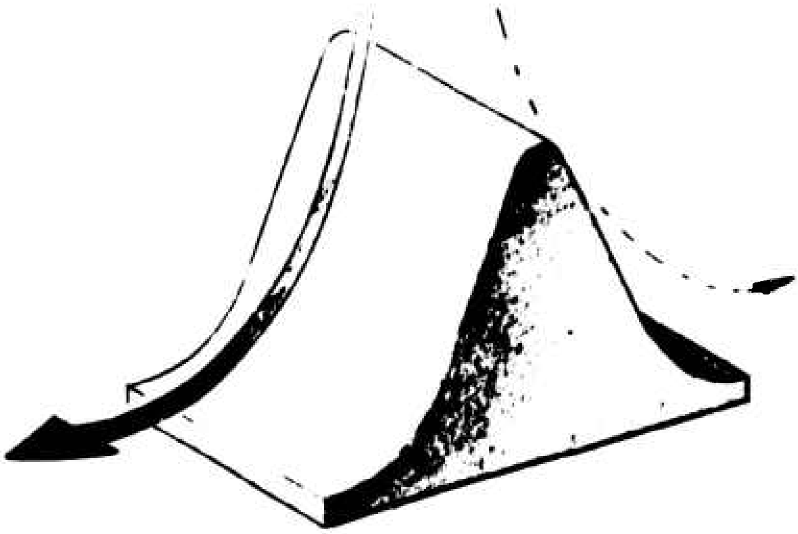
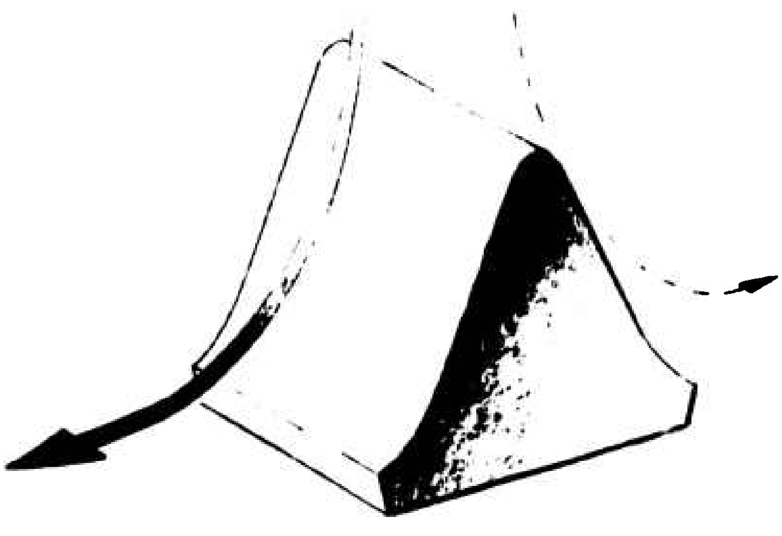


FIGURE 17: Example of Card Used for Retrieval System

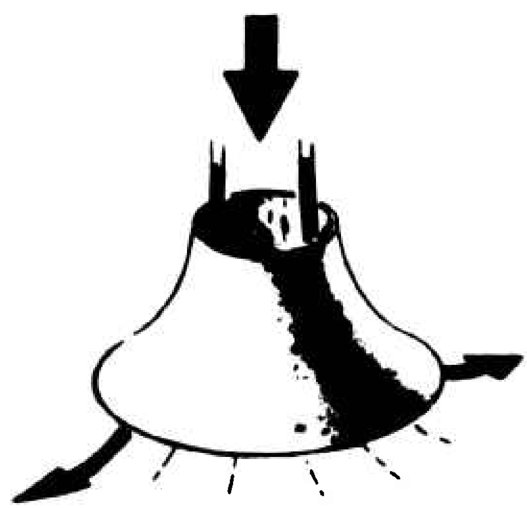


(a) Two-Way Wedge

(b) Two-Way Wedge with Auxiliary Jets



(c) Chopped Radius Wedge



(d) Coanda Effect Deflector

FIGURE 18. Possible Deflector Configurations (Martin CR-64-84 Vol. II, NAS10-1107, SPREE, Martin Company, Denver, Colorado, Dec., 1964).

This installation refers to complexes 40 and 41.

Complex 39a has a flame trench 700 feet long and 60 feet wide and 40 feet deep lined with super duty brick using tongue-and-groove construction also bonded with epoxy. About 98,000 square feet of refractory are employed in this configuration.

It should be noted that the epoxy adhesive is quite sensitive to deterioration when exposed to liquid oxygen. An area of development for refractory adhesives exists here.

In closing this section, it should be recorded that the Kennedy Space Center Complexes were used as installation examples. The literature will certainly reveal other facilities at which application examples could be found. For example, NAVWEPS Report 8300, Volume I, A Bibliography of the Vulnerability of Nuclear Weapons: Blast Environment, March 31, 1965, will reveal many related study areas. Publications relating to the fracture and breaking of rock and rock mechanics like the type supplied by U. S. Naval Ordinance Test Station, China Lake, California, reveal related application areas. Time did not permit inspection or conferences with other organizations having capability in surface blast effects like the U. S. Army Waterways Experiment Station, Vicksburg, Mississippi, or U. S. Naval Civil Engineering Laboratory at Port Hueneme, California.

VI. SUMMARY

A survey has been made of literature relating to application of refractory materials to missile blast resistance. It has included library reference sources, special abstract

refractories in these structures include high order thermal shock resistance, erosion resistance and chemical reactivity. All of these are markedly dependent on temperature and the proximity of the rocket nozzle to the impingement zone. These behavioral factors depend on intrinsic properties of the refractory but also on operational factors. The latter must be established by correlating simulative model studies with the full scale operation. Such a correlation becomes increasingly more difficult.

Hardness, density, thermal diffusivity and volume stability seem to be the intrinsic properties of the refractory upon which their behavior depends. The protection problem has been approached by using high density, high thermal diffusivity, hard materials. It has also been approached by using low density, low thermal diffusivity, materials which have predictable ablation or erosion behavior.

The first approach may utilize manufactured shapes. The second approach may use a refractory concrete with a calcium aluminate cement bond, but used in a temperature range where ablative protection results from cement-aggregate reaction.

Solid fuel rockets introduce high velocity particle impingement and thermal radiation to the surroundings as conditions of operation. As the thrust to weight ratio decreases, these factors become paramount.

VII. RECOMMENDATIONS

The survey indicates need for a continuing effort in the study of utilization of refractories during all levels of

planning. The total refractory system will not be composed of one all purpose material, but rather a system of materials, each performing its special function. Evaluation of refractory performance should be improved to include the study of launch facility performance. Even after the blast impingement is contained and directed in a controlled path, the problem of dissipating heat along this path will be a situation requiring refractory materials design criteria in addition to the normal concept of concrete structural design.

Commercial refractories manufacturers and engineering firms have broad experience in situations requiring this design concept. They should play an important part in future programs. Property measurement should be deemphasized and evaluation of behavior should be stressed. The broad knowledge of refractories materials science should be translated as rapidly as possible to this utilization-evaluation stage to provide feedback for materials development.

APPENDIX

VIII. APPENDIX

A. Retrieval System

The method used for the storing and retrieving of the assembled references is a modification of the system devised by the American Society for Metals. The literature compiled has been categorized according to the topic outline shown below.

A. MATERIALS AND THEIR PROPERTIES

1. Commercial Refractory Materials
   a. Shapes
      (A) Fireclay
      (B) High-Alumina
      (C) Basic
      (D) Insulating
      (E) Silicon Carbide
      (F) Carbon Based
      (G) Zirconia Based
      (H) Silica
      (I) Other

   b. Monoliths
      (A) Plastics
      (B) Ramming Mixes
      (C) Castables
      (D) Gun Mixes
      (E) Other

   c. Jointings
      (A) Organic Based Adhesives
      (B) Inorganic Mortars
      (C) Others

   d. Granular
      (A) Sintered
      (B) Melt Cast

   e. Other

2. Developmental Materials
   a. Oxides
   b. Carbides
   c. Borides
   d. Nitrides
   e. Silicides
   f. Sulfides
   g. Aluminides

h. Organics

i. Other

3. Refractory Coatings

4. Ablative Materials

5. Other

B. EFFECT OF PROCESS VARIABLES ON CRITICAL MATERIAL CHARACTERISTICS

1. Thermal Shock
   a. Composition
   b. Microstructure
   c. Fabrication
   d. Heat Treatment
   e. General

2. Erosion Resistance
   a. Composition
   b. Microstructure
   c. Fabrication
   d. General

3. Reactivity
   a. Composition
   b. Microstructure
   c. General

C. MATERIALS UTILIZATION

1. Materials Testing

2. Materials Specification

3. Materials Application

4. Miscellaneous

By use of the topic headings in the outline, all references pertaining to a specific subject can be retrieved from the card system.

The retrieval system uses a card with three rows of holes across the top and bottom, and two rows on each side. Figure 17 is an illustration of the card. Only the three rows across the top are presently utilized in this system; however, if

there is a desire in the future to index the references in some other manner, (such as by author) the other sides of the card can be utilized.

The three rows across the top have been divided in half with the left hand side being used for the first, second, and third orders of the outline and the right hand side for fourth, fifth, and sixth. Only the first four orders are being utilized in the outline presently; but if in the future, some of the fourth order terms such as fireclay, castables, etc. require further subdivision, they can easily be categorized into the fifth and possibly the sixth order.

After a reference has been classified under the appropriate heading, its abstract was mounted on the retrieval card which was then coded to give the proper identification for retrieval. The source of the abstract was also indicated on each card.

Assume a reference was classified under nitrides in the outline.

A. MATERIALS AND THEIR PROPERTIES

2. Developmental Materials
d. Nitrides

The entire column of three holes over capital A would be punched to signify that the reference belonged under A. MATERIALS AND THEIR PROPERTIES; the top two holes over number 2 would be punched to show that it belonged under 2., Developmental Materials, and the top hole over small d would be punched to show that it belonged under d., Nitrides.

If a reference is categorized so that its first, second, and third orders or a combination of any two lie in the same column of capital letter, number, and small letter, then the two columns marked "DUP" on the left-hand side must be utilized. An example would be all references listed under fireclay.

A. MATERIALS AND THEIR PROPERTIES

1. Commercial Refractory Materials
a. Shapes
(A) Fireclay

Notice that capital A, number 1, and small a all lie in the same column, and consequently when the entire column is punched to indicate that the reference belongs under first order A., MATERIALS AND THEIR PROPERTIES, the second and third orders in that column cannot be used. To indicate the reference belongs under second order, number 1, the top two holes in the column marked (1st) on the left-hand side are punched, and the top hole in the column marked (2nd) is punched to show that the reference belongs under third order, small letter a. The entire column over capital A on the left is also punched to show that the reference belongs under fourth order, capital (A) Fireclay.

The top hole in the "DUP" column marked (2nd) is always punched when the third order small letter appears in the same column with either the first order capital or second order number, or both simultaneously. For example, a reference under oxides would have the entire column over capital A punched, indicating MATERIALS AND THEIR PROPERTIES; the top holes in

the column over number 2 to indicate Developmental Materials, and the top hole in the (2nd) column on the left-hand side to show the reference belonged under small a., Oxides. The "DUP" column on the right-hand side of the card can be utilized in the same manner if the outline is ever expanded to the fifth and sixth orders.

To retrieve all references pertaining to a particular topic in the outline, needles would be placed through the appropriate series of holes; the cards would then be lifted, and all references on that subject would drop out of the card pack. To retrieve all references pertaining to borides, a needle would be placed in the bottom hole in the column over first order, capital A to indicate MATERIALS AND THEIR PROPERTIES; in the middle hole in the column over second order number 2 to show Developmental Materials, and in the top hole in the column over third order, small letter c to indicate Borides. The cards are then lifted by use of the needle, and all references pertaining to borides will drop from the card pack. If the topic is in the fourth order category such as fireclay, high-alumina, etc., a fourth needle would be placed in the appropriate bottom hole of a column on the left to indicate the particular fourth order capital letter (A), (B), (C), etc. The needles are also placed in the appropriate holes in the two "DUP" columns on the left when a combination of the first order capital letter, second order number, and third order small letter are in the same column

B. Acknowledgement of Survey Participants

Approximately 200 questionnaires were mailed to individuals or agencies representing: (1) refractories manufacturers; (2) government agencies and departments known to be doing work directly on blast resistant materials; (3) agencies doing work in related areas of blast effects; (4) industrial laboratories in which work relating to general refractories utilization was known to exist. The Directory of The Refractories Institute was used to establish the mailing list for refractories manufacturers.

Although the survey may not have been completely comprehensive, it provided a preliminary source of significant information to add to the literature retrieval obtained through the usual channels of abstract publications, textbooks, abstract services and other library facilities. In fact, many of the key reports were obtained only through this survey questionnaire.

Listed alphabetically below are those sources which provided specific information. Many others replied to the questionnaire but supplied no tangible information.

Aluminum Company of America
2900 Missouri Avenue
East St. Louis, Illinois
H. M. Hartle
Product data on CA-25 calcium aluminate cement, calcined alumina, and tabular alumina.

The Chas. Taylor Sons Co.
Cincinnati, Ohio
Harold D. Prior
Property data on high alumina monoliths.

Commercialores, Inc.
Clover, South Carolina
J. E. Castle
Property data on kyanite.

Douglas Missile and Space Systems Division
Santa Monica, California
A. B. Eisenberg
Marquardt Corp. Report FE-223-1, 7/60, "Feasibility Study Report from Dry Refractory Flame Deflector Materials."

George C. Marshall Space Flight Center
Huntsville, Alabama
O. K. Duren
Martin Denver Report No. IR-65-1, "Solid Propellant Rocket Exhaust Effects (SPREE) and Methods of Attenuation" - Phase II Report Summary.

Glasrock Products, Inc.
1101 Glidden Street
Atlanta, Georgia
Cecil Mason
Martin Company, 624-A, "(Titan III) Blast Materials Evaluation."
Martin Company, 624-A, "Exhaust Duct Lining (Titan III)"
Martin Company, "Pershing Blast Deflector Materials Development."
Martin Company, "The Performance of Fused Silica in a Program Supersonic Rocket."

A. P. Green Fire Brick Co.
Mexico, Missouri
J. Denton
Detailed information on material specifications and installation of refractories at Kennedy Space Center launch complexes. Extended invitation for personal conference, accepted May 25, 1965.

General Electric Missile and Space Division
Space Sciences Laboratory
Valley Forge Space Technology Center
Philadelphia, Pennsylvania
L. R. McCreight, W. H. Sutton, H. Rausch
Information on ablative materials, composites and fiber technology survey.

Harbison-Walker Refractories Co.
2 Gateway Center
Pittsburgh, Pennsylvania
Thomas W. Smoot
General property data on silica, fireclay, and castable refractory materials.

International Pipe and Ceramics Corporation
2901 Los Felix Boulevard
Los Angeles, California
Roger R. Riley

Description of ASTM Abrasion Test - ASTM C-8 Committee, Section III M, property data on high alumina monolith.

R. B. Judson
21 Grafton Street
Chevy Chase, Maryland

Vitro Laboratories Technical Note TN01530-1, "Interim Report on the Effects of Rocket Blast on Hawk Fixed Installations."

Resco Products, Inc.
Norristown, Pennsylvania
C. Robert Enoch

National Bureau of Standards Project 0903-21-4428 "Investigation on Aggregates and Concretes Used in Rigid Pavements Subjected to High and Fluctuating Temperature."

URS Corporation
1811 Trousdale Drive
Burlingome, California
William L. Durbin

Abstracts on the effects of shock waves on materials.

U. S. Air Force Rocket Propulsion Laboratory
Edwards, California
William Lawrence

Marquardt Rept. FE 223-1, 7/60, "Feasibility Study Report for Dry Refractory Flame Deflector Materials."
Marquardt Rept. FE 223-3, 7/61, "Design Criteria Report - Uncooled Refractory Lined Flame Deflector."
Marquardt Rept. FE 223-4, 8/61, "Final Report - Uncooled Refractory Lined Flame Deflector."

U. S. Army Engineers, Ohio River
Corps of Engineers
Ohio River Division Laboratories
Cincinnati, Ohio
B. U. Duvall

Extensive file covering work dating back to January, 1947; frequent conferences including Kennedy Space Center facilities inspection.

U. S. Army Engineer Waterways Experiment Station
Vicksburg, Mississippi
Jerome J. Krochmal

"Capabilities in Surface Blast Effects Research." Extended invitation for conference.

U. S. Naval Civil Engineering Laboratory
Port Hueneme, California

Technical Report R-089, 11/14/60, "Effects of Jet-Engine Exhaust on Virginia Diabase Concrete Pavement."

Technical Report R-170, 11/6/61, "Effect of Aggregate Size on Thermal Shock Resistance."

Technical Report R-169, 11/22/61, "Effect of Temperature Rise on Compressive Strength of Hardened Cement Paste."

U. S. Naval Ordinance Test Station
China Lake, California
Carl F. Austin

Publications on the effect of shock penetration on rock and other brittle materials.

U. S. Naval Weapons Evaluation Facility
Kirtland Air Force Base
Albuquerque, New Mexico
T. Kelly

"Bibliography on the Vulnerability of Nuclear Weapons."

Vitro Laboratories
14000 Georgia Avenue
Silver Springs, Maryland
Robert E. Grissett

Vitro Lab Technical Note TN-1530-1, "Interim Report on the Effects of Rocket Blast on Hawk Fixed Installation."

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Aldred, F. J., "Alumina Ceramics," Trans. Brit. Ceram. Soc., 61 (11) 705-23 (1962).
Eng. Index Service, No. 63-2884

Allen, A. C., "Nuclear and Space Ceramic Research," Ceram. Ind., 83 (4) 31-43 (1964).
Eng. Index Service, No. 64-40841

Allen, K. K., "How to Use Grog with Clay," Ceram. Age, 73 (2) 18-21 (1959).
Eng. Index Service, No. 59-15817

Allen, Robert D., "Thermal Expansion of Synthetic Graphites at Temperature Intervals Between 80° and 2000°F.," Calif. Inst. Tech. Jet Prop. Lab. Progr. Rept., 30-20 (1959).
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Eng. Index Service, No. 56-25087

Alper, A. M., McNally, R. N., Ribbe, P. H., and Doman, R. C., "System $MgO-MgAl_2O_4$," J. Am. Ceram. Soc., 45 (6) 263-68 (1962).
Ceram. Abstr., 1962, 180f

Alper, Allen M., Begley, Edward R., Londeree, Joseph W., and McNally, Robert N., "Fused Cast Refractory and Method of Making," U. S. 3,132,953 May 12, 1964.
Ceram. Abstr., 1964, 219c

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A literature survey was conducted to study the criterion for the utilization of refractory materials for thermal protection of launch facilities. Sources were surveyed from the viewpoint of the refractories engineer including: (1) published literature relating to refractory materials in general; (2) literature relating to specific applications under simulated or actual launch conditions; (3) preliminary contacts with refractories manufacturers and engineering departments; (4) preliminary contacts with government agencies and facilities working in this or related problem areas. A historical review traced the development of the subject from the early use on aircraft runway pavement to present missile sites. Kennedy Space Center inspections and Ohio River Division Laboratories' Programs were used as examples to characterize the problems involved.

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It is highly desirable that the abstract of classified reports be unclassified. Each paragraph of the abstract shall end with an indication of the military security classification of the information in the paragraph, represented as *(TS), (S), (C), or (U)*.

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